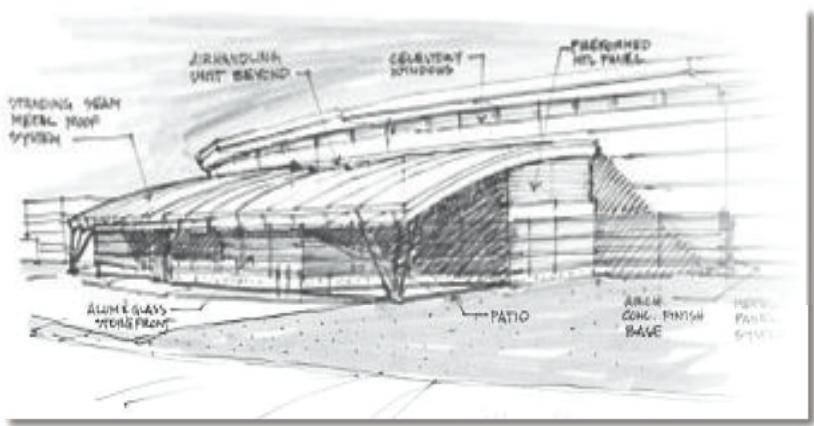
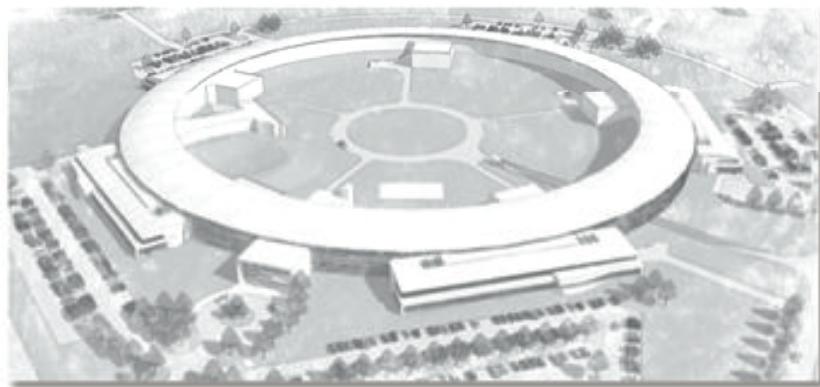


SRX: High Photon Flux / High Spatial Resolution X-ray Spectroscopy at NSLS-II



Vincent De Andrade

SRX Associate Scientist

Rock & Cell Workshop

September 17-18, 2012

SRX: Submicron Resolution X-ray spectroscopy beamline

- 1) Scope & Overview of SRX
 - Scope of the beamline
 - Layout
 - Scientific capabilities / Beamline Performances
- 2) End stations: microprobe & nanoprobe
 - Versatility
 - Sample stage / Nano-positioning
 - Detector & analytic techniques
 - Sample Environment
 - Schedule: the miles stones
-) Conclusion

Scope and overview of SRX

The team

Juergen Thieme (*group leader*)

Vincent De Andrade (*associate scientist*)

Yuan Yao (*mechanical engineer*)

Coming soon:

Niaja Farve (*PhD student*)

Carey Koleda (*technician*)

Support / Collaborators

W. Lewis (control)

O. Chubar (Wavefront simulation)

M. Idir (Optics)

R. Tappero (beam line)

P. Northrup (beam line)

K. Jones (tomography)

P. Siddons (Detectors)

A. Broadbent (Interfaces)

E. Nazaretski (Nano-positioning)

L. Reffi / A. De Santis (Ray tracings)

V. Ravindranath & S. O'Hara (FEA calculations)

L. Doom (Frontend design)

...

The Beamlne Advisory Team

Peter Eng (U of Chicago)

Jeffrey Fitts (BNL)

Chris Jacobsen (ANL)

Keith Jones (BNL)

Antonio Lanzirotti (U of Chicago)

Matt Newville (U of Chicago)

Paul Northrup (SUNY Stony Brook)

Richard Reeder (SUNY Stony Brook)

Mark Rivers (U of Chicago)

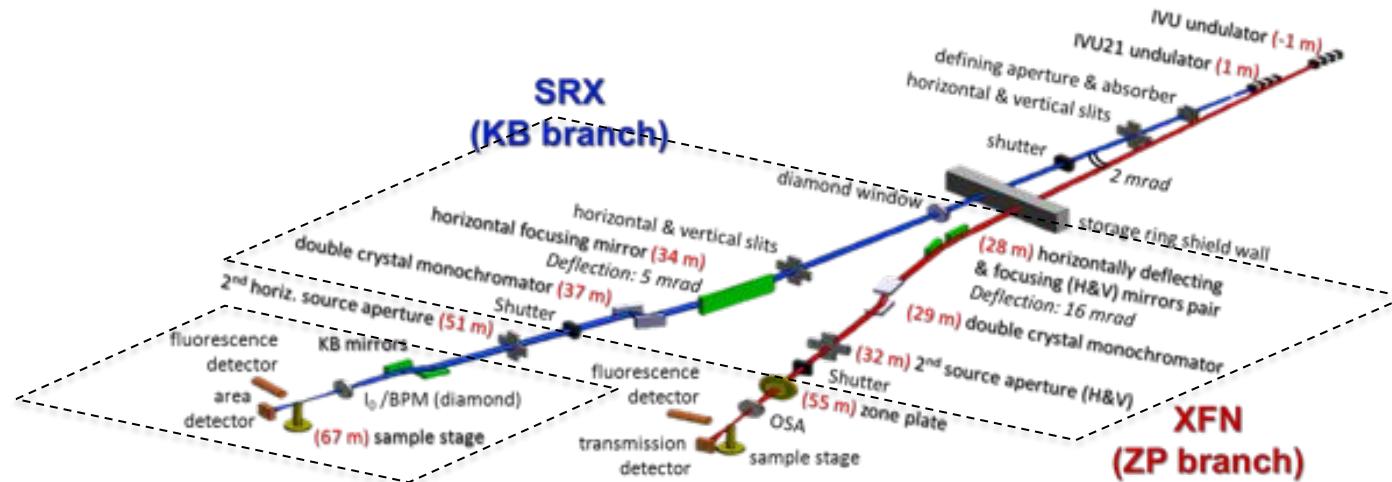
Stephen Sutton (U of Chicago)

Stefan Vogt (ANL)

Gayle Woloschak (Northwestern

University)

Scope and overview of SRX



General background

Analytical resources have to be developed to advance our understanding of complex natural and engineered systems that are heterogeneous on the micron to submicron scale

Scientific areas

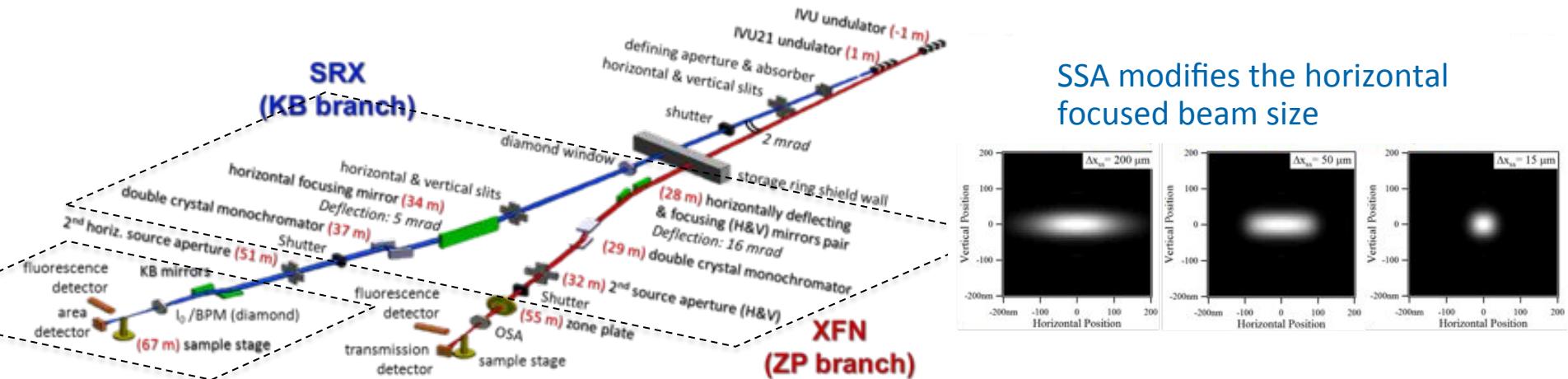
- Environmental Sciences
- Earth & Universe Sciences
- Life Sciences
- Material Sciences
- Energy research Sciences
- Cultural Heritage



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Scope and overview of SRX



SRX at NSLS-II:

- Beamline with versatile sample setup for experiments in environmental, life, and material sciences.
- Designed to allow two independent beamlines optimized to perform high flux and high spatial resolution spectroscopy
- SRX with KB mirrors optimized for $E = 4.65 - 25 \text{ keV}$. Two sets of KB optics will create either a sub- μm or sub 100 nm focal spot
- X-ray Fluorescence Nanoprobe (XFN) using zone plates is optimized for $E = 2 - 15 \text{ keV}$, focal spot $< 30 \text{ nm}$. *Not in the present scope.*

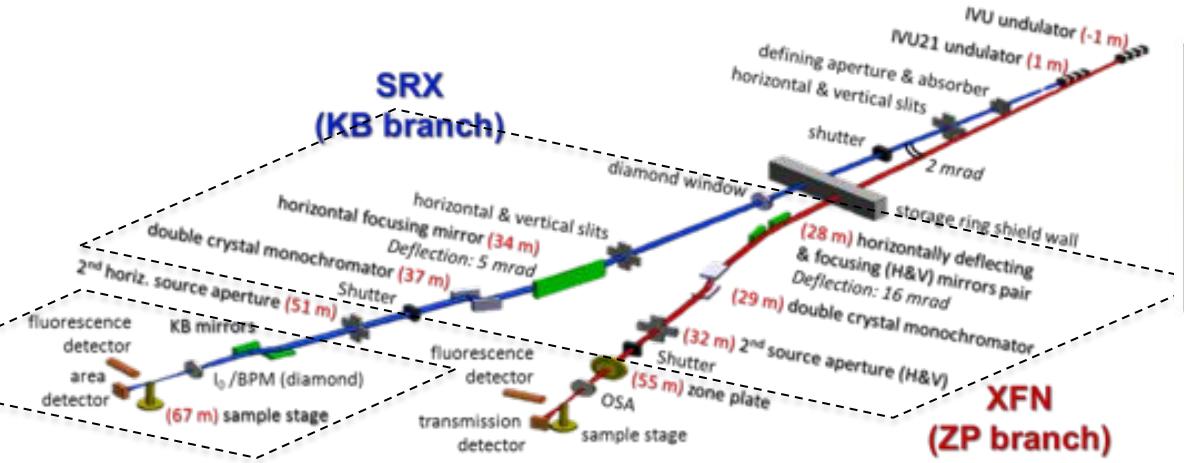
SRX Beamlne performances

- **Source:** In vacuum undulator, $I = 21 \text{ mm}$, $L = 1.5 \text{ m}$
- **Energy range:** $4.65 \text{ keV} \leq E \leq 25 \text{ keV}$
- **Monochromator:**
Horizontally deflecting DCM: Si (111) or Si (311)
- **Energy resolution:** $\Delta E \approx 1.5-2.5 \text{ eV} @ 12 \text{ keV}$
 $\Delta E \approx 0.8 \text{ eV} @ 7 \text{ keV}$
(in collimating mode) $\Delta E \approx 0.1 \text{ eV} @ 7 \text{ keV}$

Performances at the focal spot

- High flux setup: $\phi \approx 0.5 \mu\text{m}$ at $> 10^{13} \text{ phot/sec}$
- High resolution setup:
 $\phi \approx 70 \text{ nm}$ at $10^{11}-10^{12} \text{ phot/sec} @ 7 \text{ keV}$
 $\phi \approx 30 \text{ nm}$ at 10^{11} phot/sec from 12 keV

Scope and overview of SRX



H		2 keV < Absorption Edge < 4.6 keV										He
Li	Be	4.6 keV < Absorption Edge < 23 keV										
Na	Mg											
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In
Cs	Ba	Lanth.	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl
Fr	Ra	Act.	Rf	Db	Sg	Bh	Hs	Mt	Ds	Rg	Cn	Uut
												Uuq
												Uup
												Uuh
												Uus
												Uuo
La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm
Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md
												No
												Lr

Techniques available:

With a micro / nanobeam:

- μ-XANES spectroscopy (fluo & abs)
- X-ray fluorescence imaging (*mapping of trace elements*)
- X-ray fluorescence tomography
- Combination of microscopy & XES
- RIXS
- XRS

Full-field:

- μ-XANES
- μ-CT
- ultra fast μ-CT (10 ms / volume acquisition)
- phase contrast
- Holotomography

With a μm / nm beam + Array Detectors:

- X-ray μ-diffraction
- X-ray coherent imaging: ptychography
- Zoom μ-CT

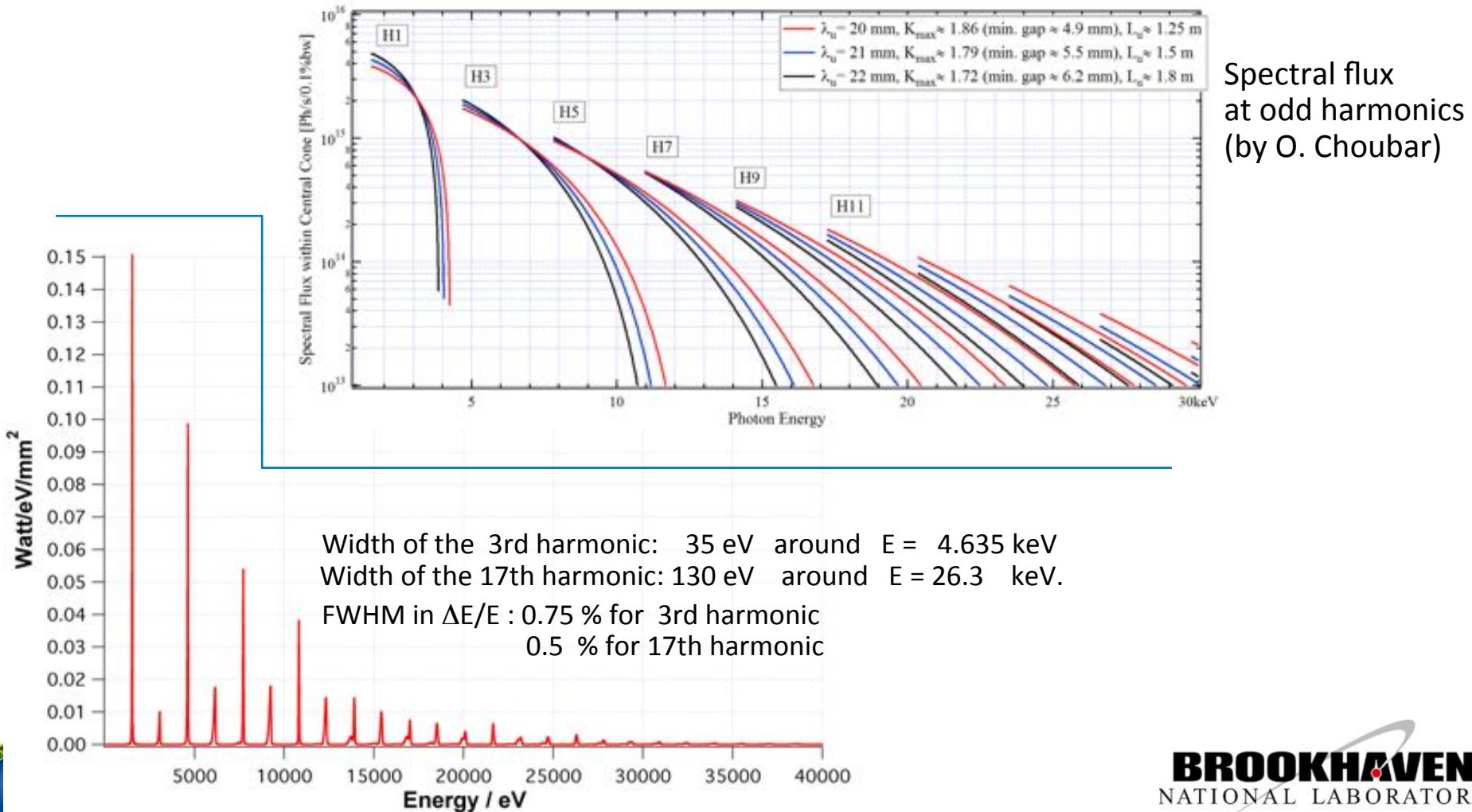


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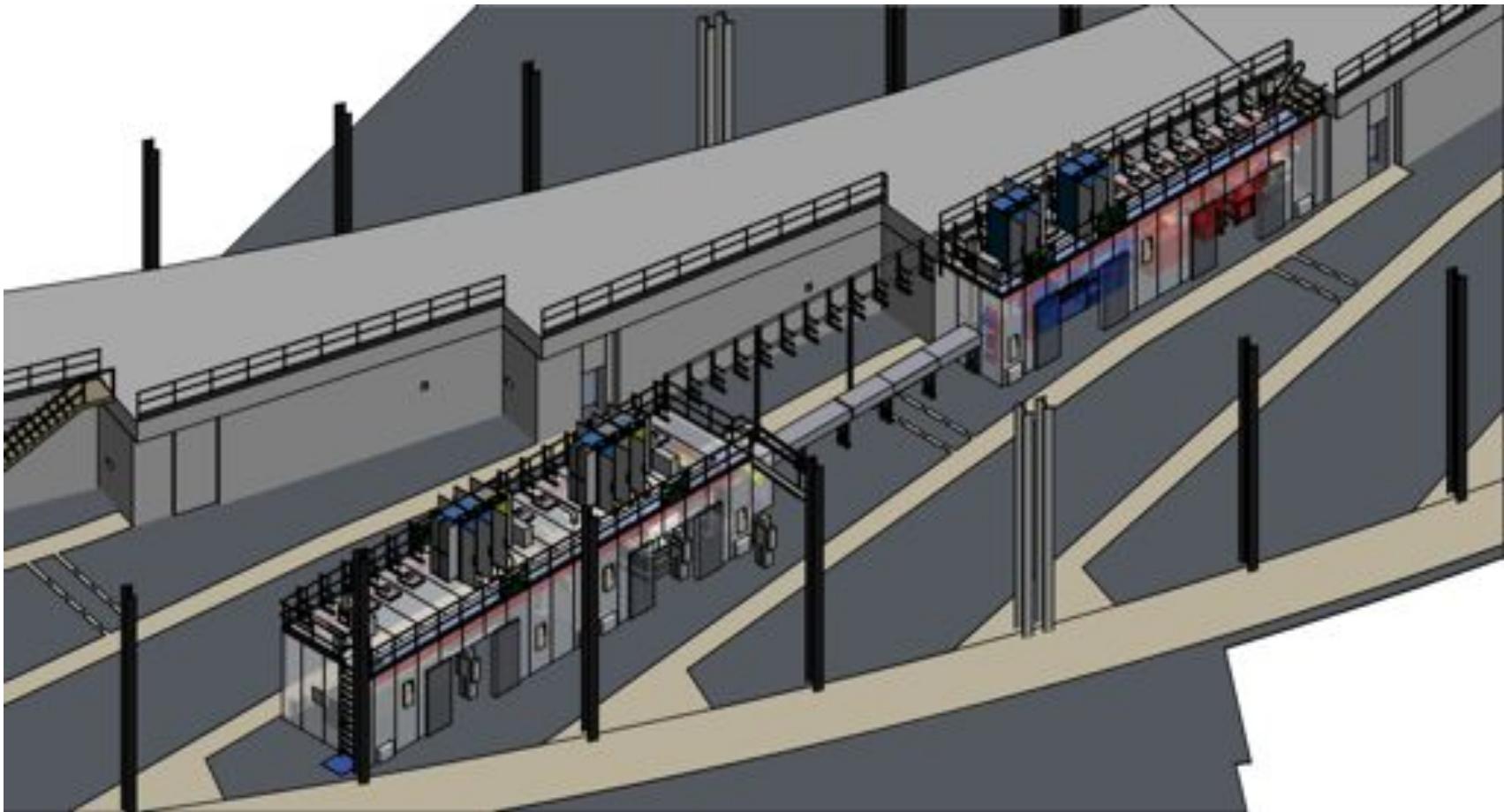
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Undulator choice

- Low beta, short straight section
- To serve both branches independently
⇒ two undulators, canted design



Overall Layout



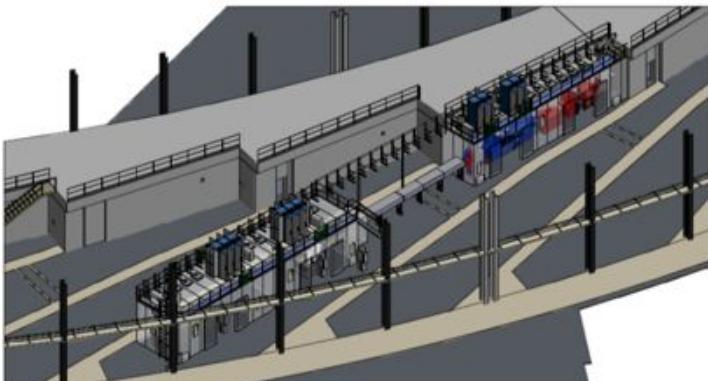
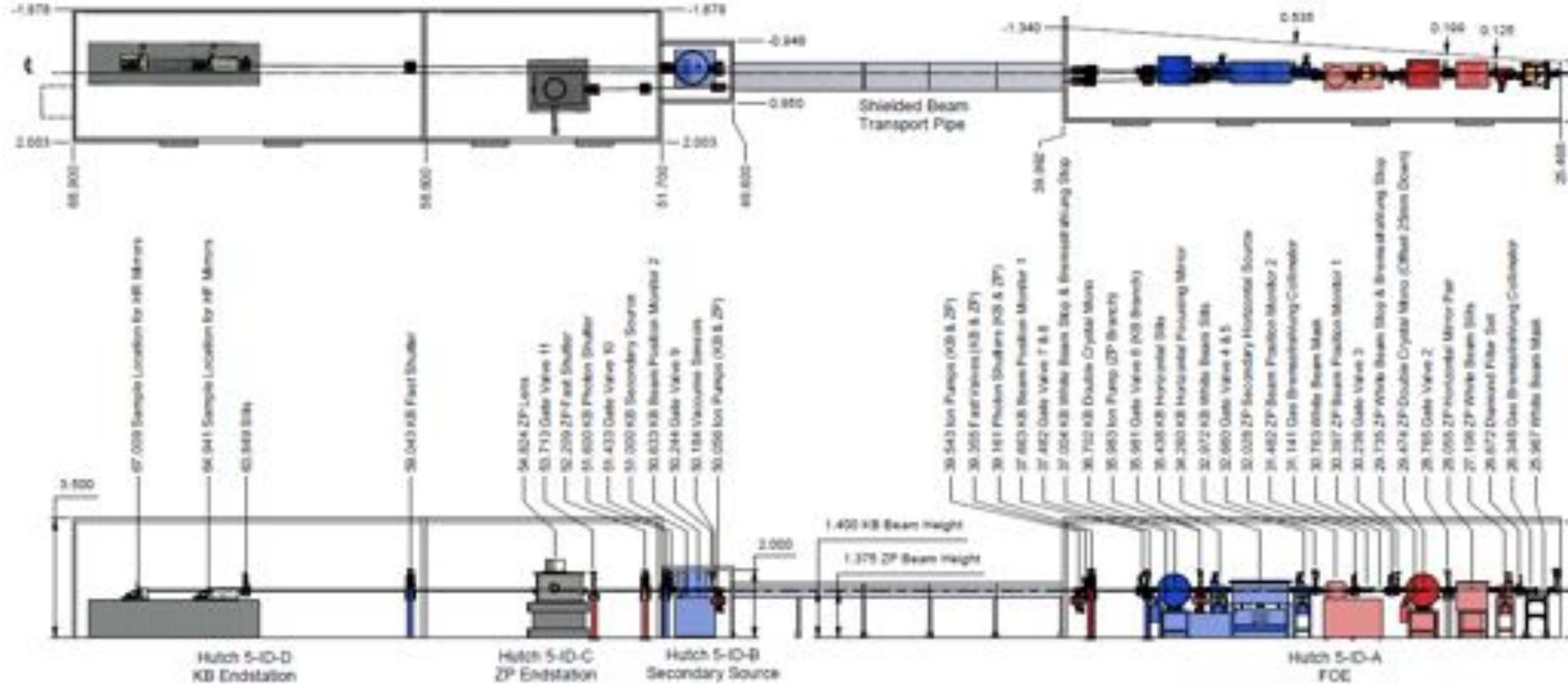
Hutch 5-ID-A (lead): Focussing mirrors, monochromators, beam defining slits for ZP branch.
Beam defining slits for KB branch in small steel hutch (5-ID-B). Steel hutes: Endstations for
ZP- (5-ID-C) and KB-branch (5-ID-D). Optical components are shining through the walls.



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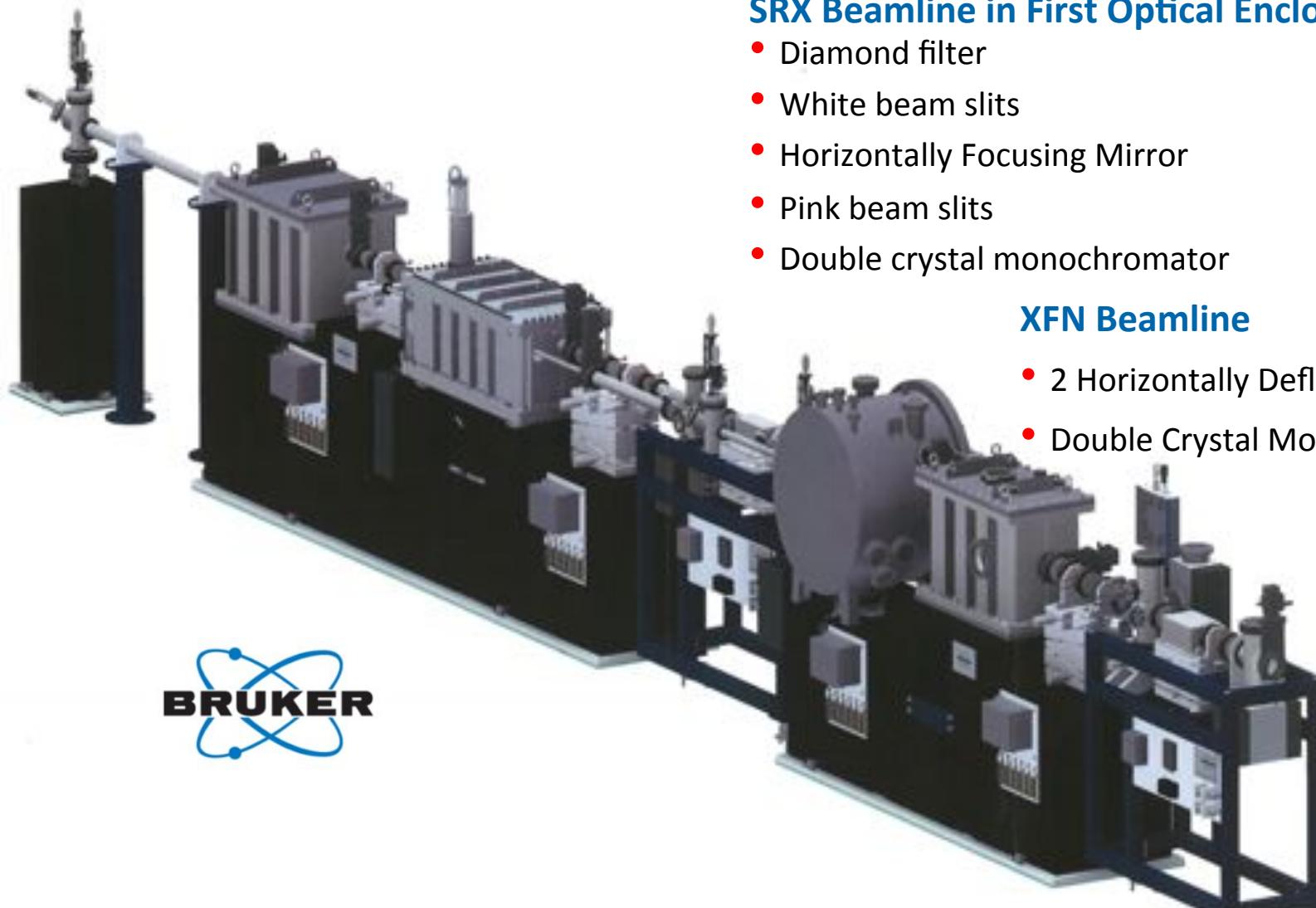
Overall Layout



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Overall Layout



SRX Beamline in First Optical Enclosure

- Diamond filter
- White beam slits
- Horizontally Focusing Mirror
- Pink beam slits
- Double crystal monochromator

XFN Beamline

- 2 Horizontally Deflecting Mirrors
- Double Crystal Monochromator



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Strategy for a high stability in the FOE

✓ Our Leitmotiv: the stability

- Horizontally focusing mirror → the gravity does not increase the pitch vibrations
- Horizontally diffracting Double Crystal Monochromator → idem
- Critical optics sharing the same granite table
- Use of a secondary source in the horizontal plane
 - *it involves to get a very stable pair of slits (SSA)*
 - *BPM at the SSA for closed loop operations with the 2nd DCM crystal*

DCM = master piece of the Beamline

- <50 nrad (1σ) vibrational stability
- Compactness for stability purpose
- Pitch of both crystals on flexures
- Cryogenic Indirect or Cu braid cooling available
- 1 full XANES in 2-3 seconds

DCM Bruker's design removed for confidentiality reason

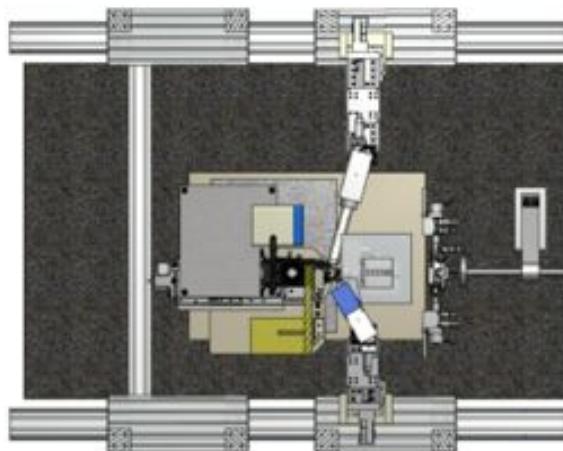


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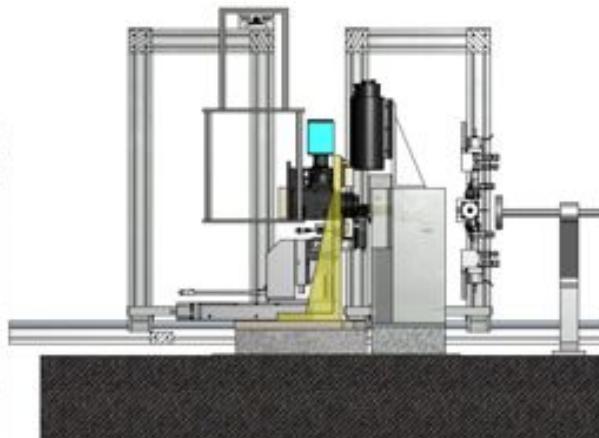
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End-station concept with 2 modes

Top view



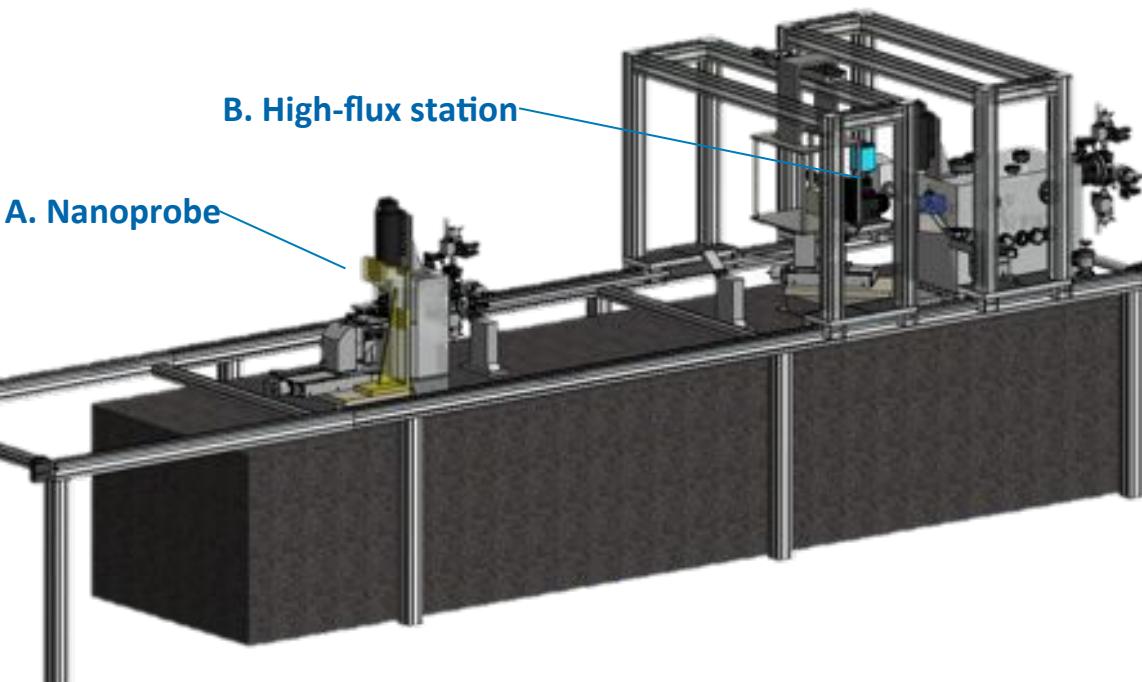
Side view



KB optics concept

Two sets of KB mirrors:

- **A: nanoprobe**
fixed curvature
short extension
short focal lengths
resolution ~ 50 nm
- **B: high-flux station**
fixed curvature
large aperture
long working distance
resolution ~ 1 to 0.15 μm



A stays fixed and aligned
B mirrors goes in & out.

Detectors move quickly around.

Science Capabilities

- Flux of more than 10^{13} ph/s in a sub-micron sized focal spot => dwell time per pixel well below 1 ms for fluorescence =>
 - fast scanning of large images, mm sized e.g. $5 \times 5 \text{ mm}^2$, $1 \mu\text{m}$ spot, 0.5 ms dwell/point in 3.5 hours (dwell time 0.1 sec => 29 days at standard conditions), image size necessary for representative sample size, e.g. energy storage
 - SRX could boost X-ray fluorescence tomography & μ -XANES tomography with a pencil beam
 - New possibilities with studies looking at trace elements (Earth Sciences)
- Intense 50 nm large monochromatic beam
 - Spatial resolution sufficiently high for imaging in situ e.g. micro- and nanoparticles, colloids in fluid cells, biofilms and microbial suspensions
 - Unmatched resolution with a monochromatic beam: enable spectroscopy with a nanoprobe. Study in situ e.g. rock weathering, oxidation/reduction of minerals, integration, storage and release of heavy metals and radionuclides, crevices, grain or phase boundaries

Name	Energy range / keV	Spot size / μm^2	Energy resolution	Photon flux in spot	Spectroscopy
SRX @ NSLS-II	4.7 – 23	0.8 x 0.7 (KB) 0.05 x 0.05 (KB)	1.5 x 10^{-4} 1.5 x 10^{-4}	> 10^{13} @ 12 keV 10^{11} - 10^{12} @ 12 keV	Y Y
NINA @ ESRF	11.2/17/33.6 keV (NI) 5-70 NA	0.01 x 0.1 (NI) (MLL) 0.05 x 1 (NA) (KB)	10^{-2} 10^{-4}	2.5×10^{12} 1.8×10^{11}	N Y
In-situ Nanoprobe @ APS	4-30	0.05 x 0.05 (KB) 0.02 x 0.02 (ZP)	10^{-2} 10^{-4}	8.7×10^{11} @ 10 keV 2.2×10^9 @ 10 keV	N Y
Nanoscopium @ SOLEIL	5-20	0.1 x 0.1 (KB)	10^{-4}	1.4×10^{10} @ 5 keV	Y





End-station presentation

- State of the Art Kirkpatrick-Baez (KB) focusing optics
- 2 versatile End-stations
- Sample stage and nanopositioning
- Detectors & analytic techniques
- Sample environment

Design of the High Flux KB system

✓ WinlightX Design

In-house piezo driven flexure
base stages

KB's WInlightX design removed for confidentiality reason



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SRW Partially-coherent wavefront propagation simulation with perfect optics

✓ Nanoprobe

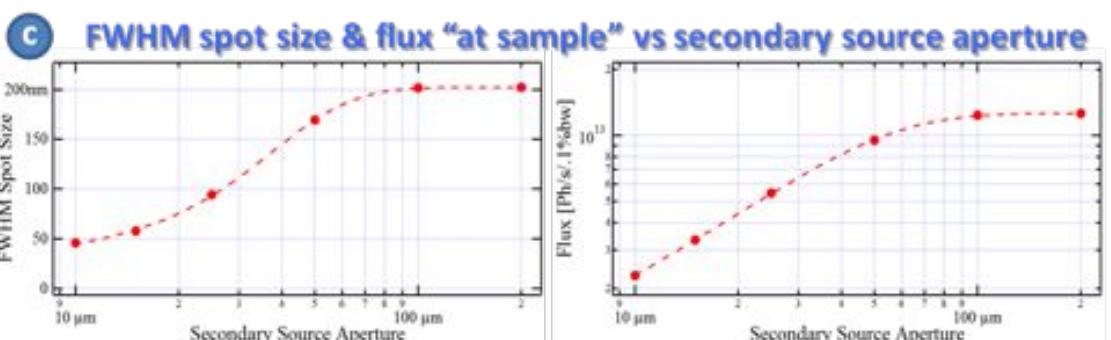
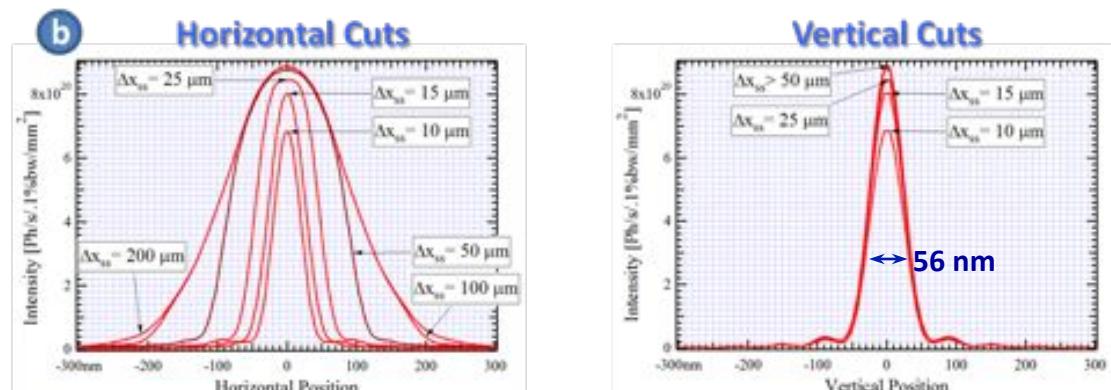
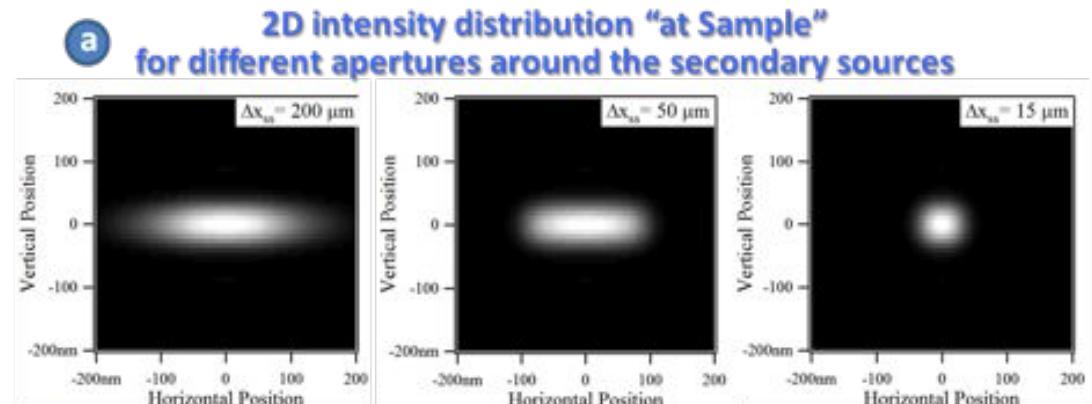
- Diffraction limited focal spot
- Fully (vert.) and partially (horiz.) coherent beam
- ray tracing useless to specify the optics

Thin elements approximation:

Assumption a KB mirror behaves like a lens.

Focal spot @ 7.2 keV:

- Vert. ~ 56 nm
- Horiz. ~ 56 nm with 15 μm SSA
- Flux ~ 10^{12} ph/s

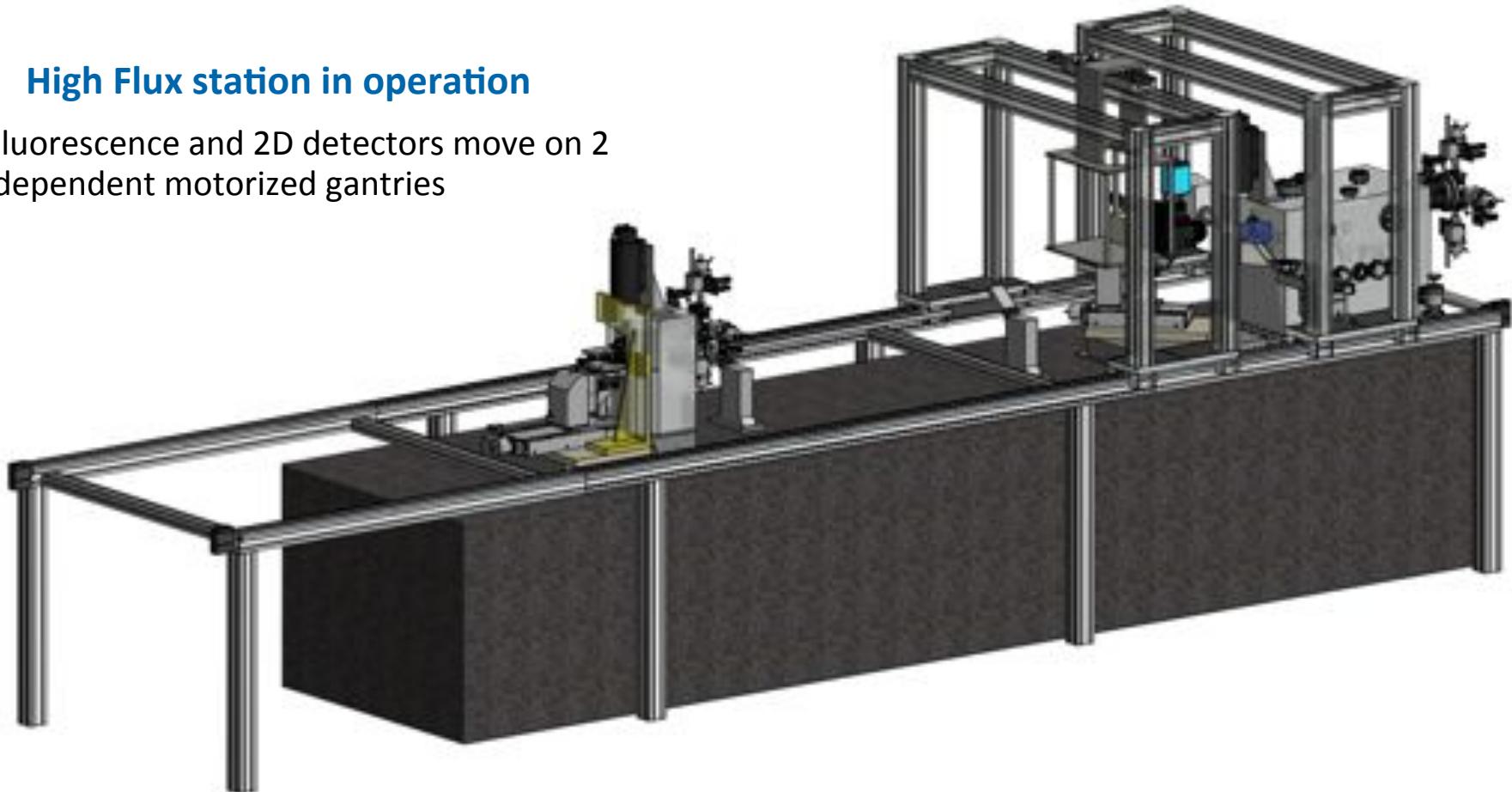


(by Oleg Chubar)

Versatility

- ✓ Goals: not either the microprobe or the nanoprobe but both at your convenience
- ✓ No need to waste half a day to switch from the microprobe to the nanoprobe
- **High Flux station in operation**

- Fluorescence and 2D detectors move on 2 independent motorized gantries

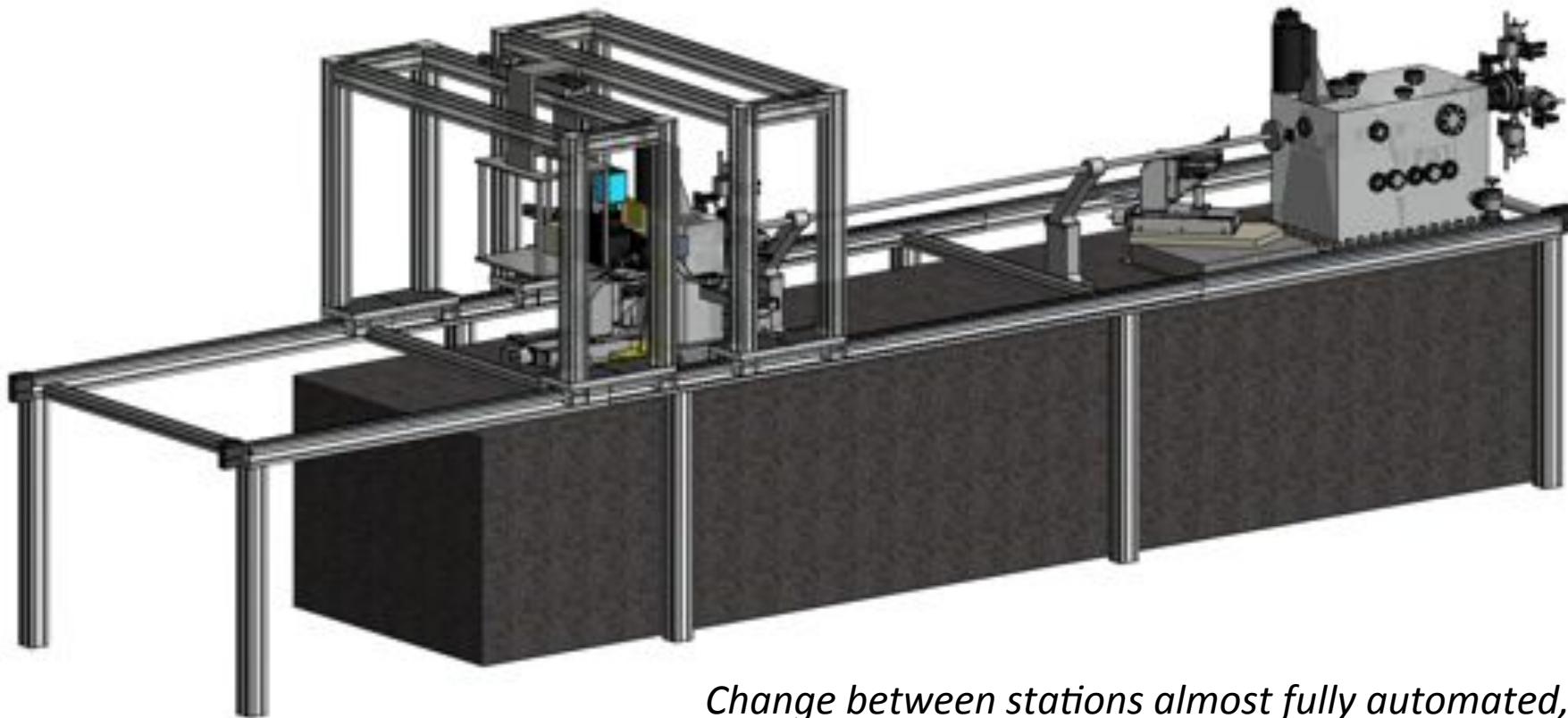


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Versatility

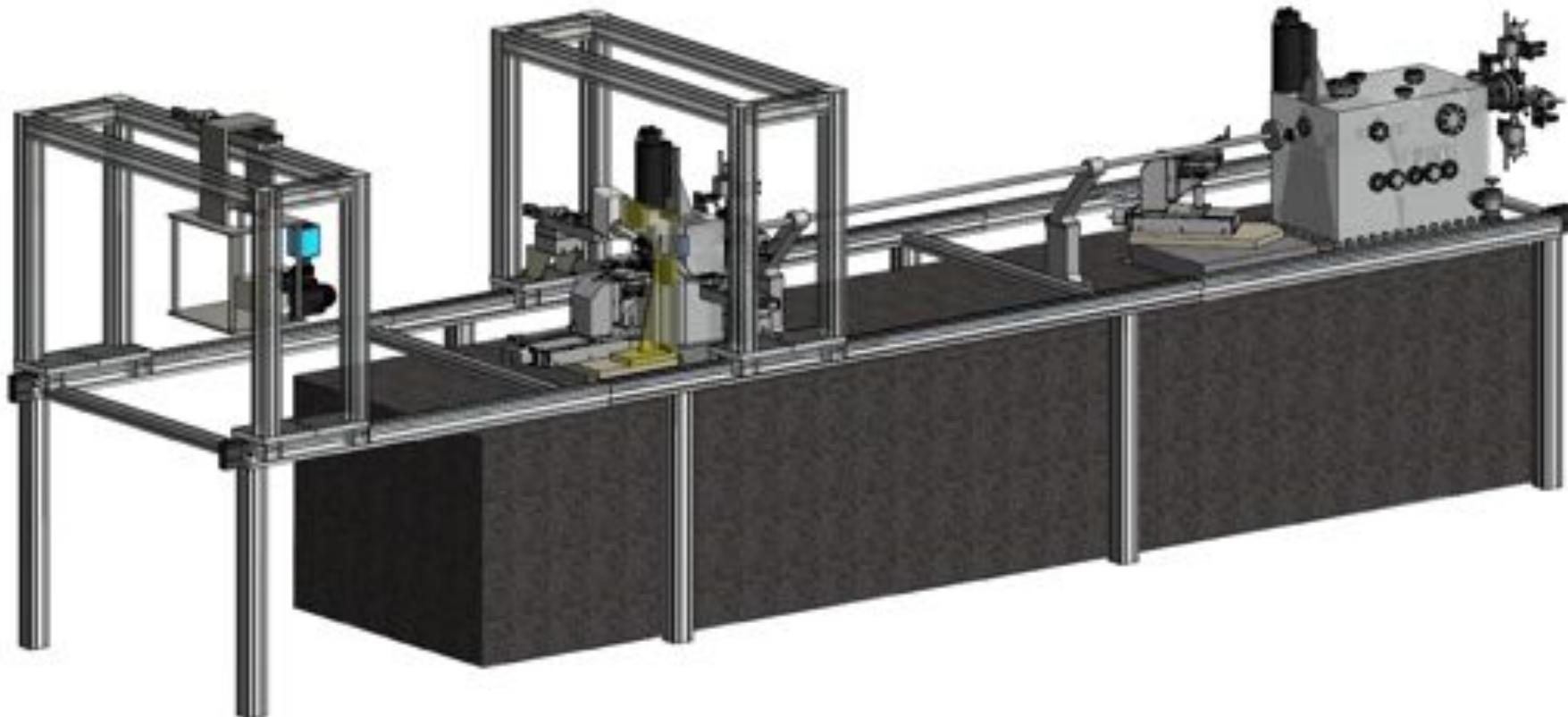
- ✓ Goals: not either the microprobe or the nanoprobe but both at your convenience
- ✓ No need to waste half a day to switch from the microprobe to the nanoprobe
- Nanoprobe in operation



*Change between stations almost fully automated,
only the sample and the tomo setup have to be
manually transported*

Versatility

- ✓ Goals: not either the microprobe or the nanoprobe but both at your convenience
- ✓ No need to waste half a day to switch from the microprobe to the nanoprobe
- **Nanoprobe in operation**
 - Geometry for ptychography, Zoom tomography

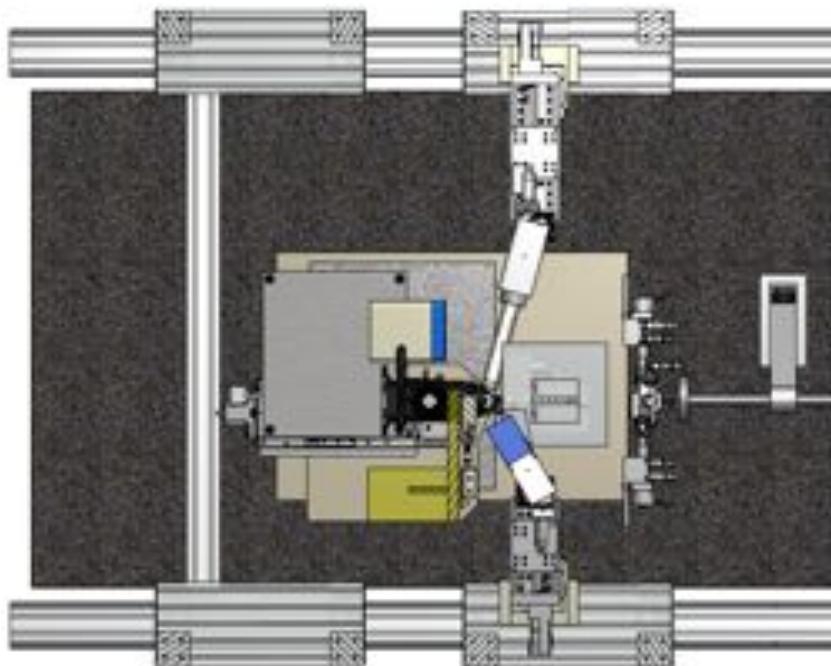


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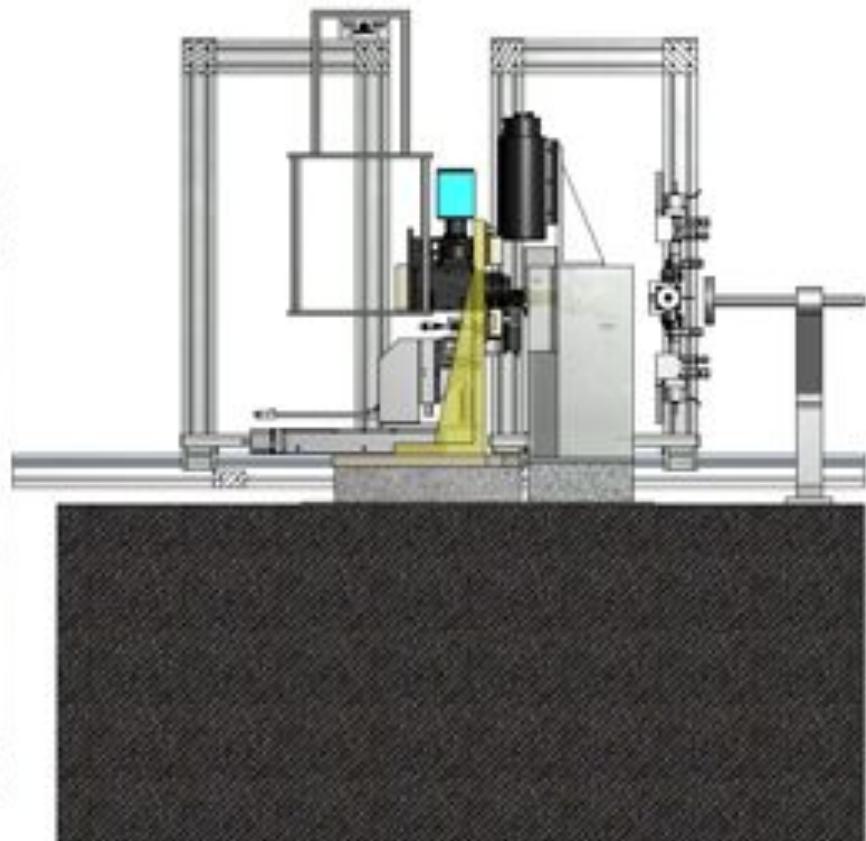
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End-station concept with 2 modes

Nanoprobe top view



Nanoprobe side view



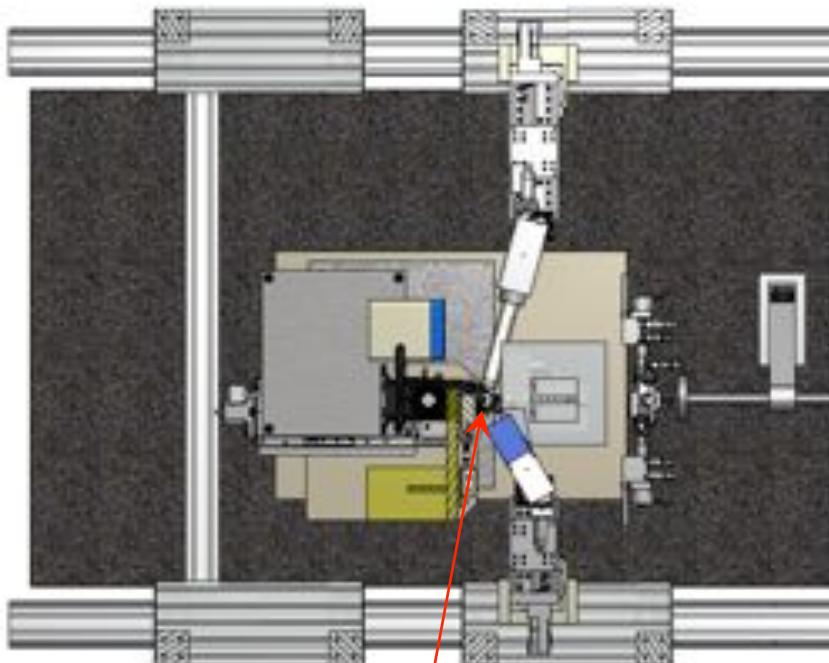
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End-station concept with 2 modes

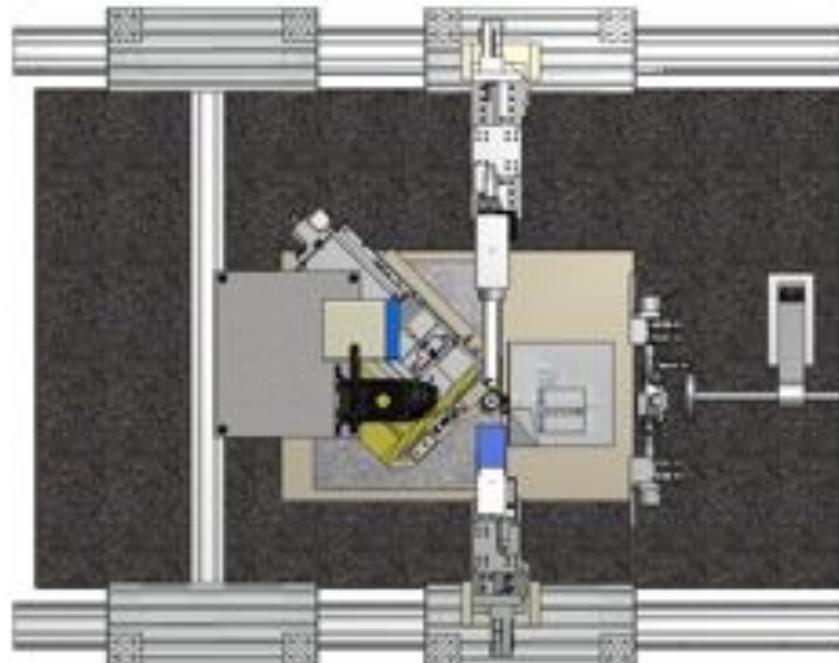
- Sample geometry @ 90°

→ minimize the beam footprint on the sample



- Sample geometry @ 45°

→ minimize the scattering on detectors



Axis of rotation of the base plate centered on the sample

→ *interferometers are carried with the base plate*



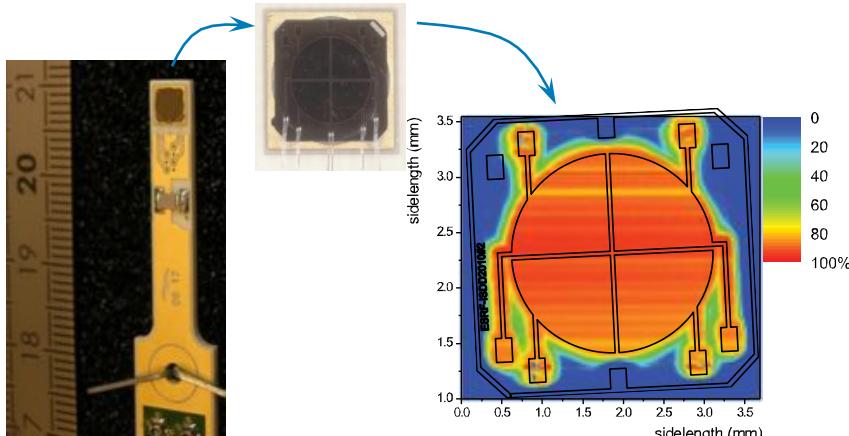
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Strategy for a high stability in the End-Station

✓ Our Leitmotiv: the stability

- Final focusing optics:
 - Long KB mirrors: no benders
 - nanoprobe: fixed curvature, short & stiff mirrors, minimization of freedom degrees
- High resolution / high throughput Diamond BPM (downstream the KB's)



J. Morse data courtesy (ESRF)

- ✓ Flux linearity demonstrated over **11 orders of magnitude**
- ✓ Position resolution ~20 nm possible for small, intense beams
- ✓ Position, timing and flux recorded for every bunch at 6.5 MHz

Data from J. Smeidley (BNL)



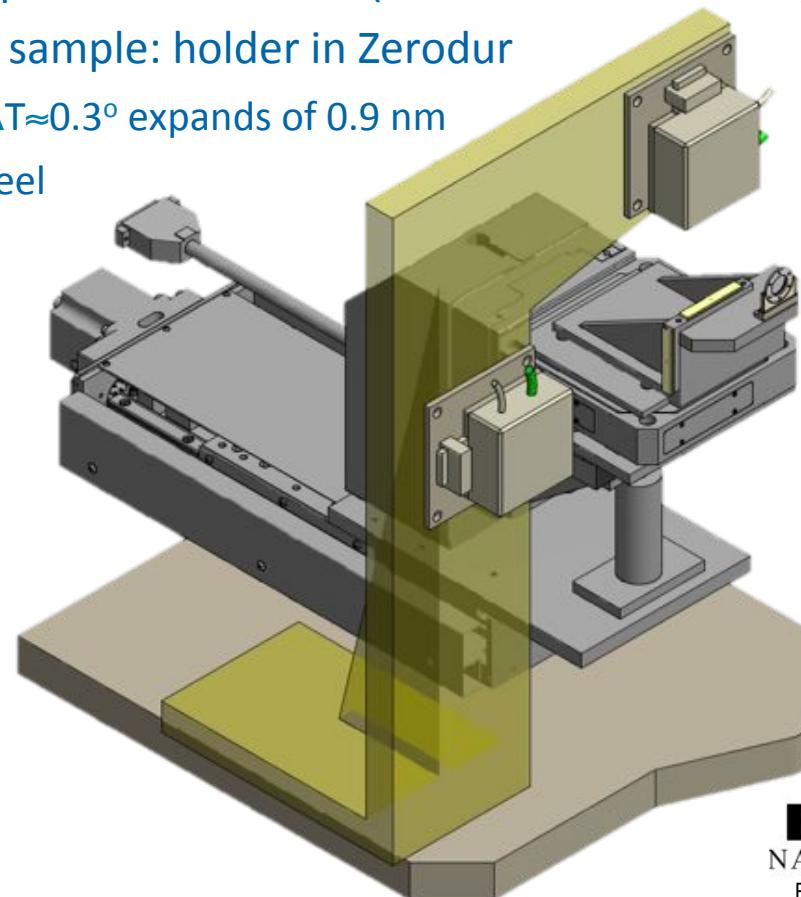
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- Final focusing optics:
 - Long KB mirrors: no benders
 - nanoprobe: fixed curvature, short & stiff mirrors, minimization of freedom degrees
- High resolution / high throughput Diamond BPM (downstream the KB's)
- Interferometers surveying the sample: holder in Zerodur
 - 15 cm of Zerodur undergoing $\Delta T \approx 0.3^\circ$ expands of 0.9 nm against 54 and 540 nm for Invar & steel



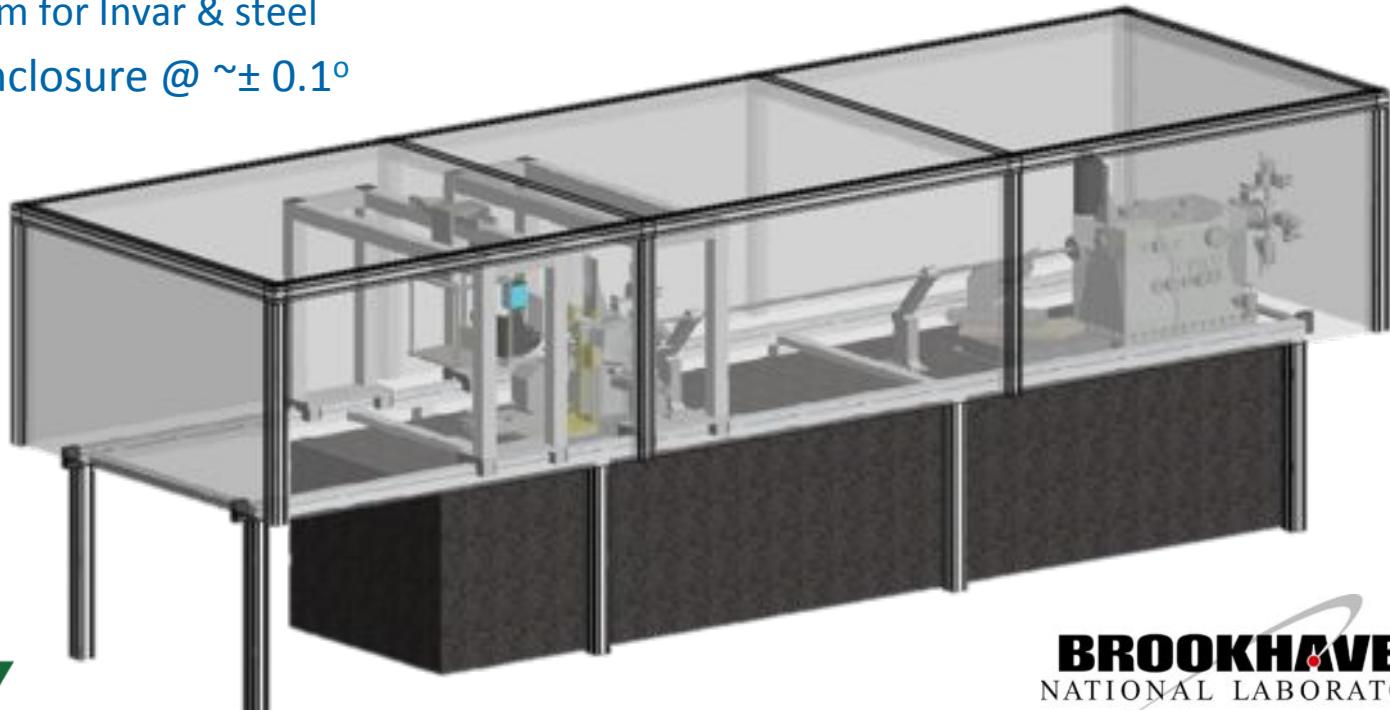
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- Interferometers surveying the sample: holder in Zerodur
 - 15 cm of Zerodur undergoing $\Delta T \approx 0.3^\circ$ expands of 0.9 nm against 54 and 540 nm for Invar & steel
- T° controlled enclosure @ $\sim \pm 0.1^\circ$



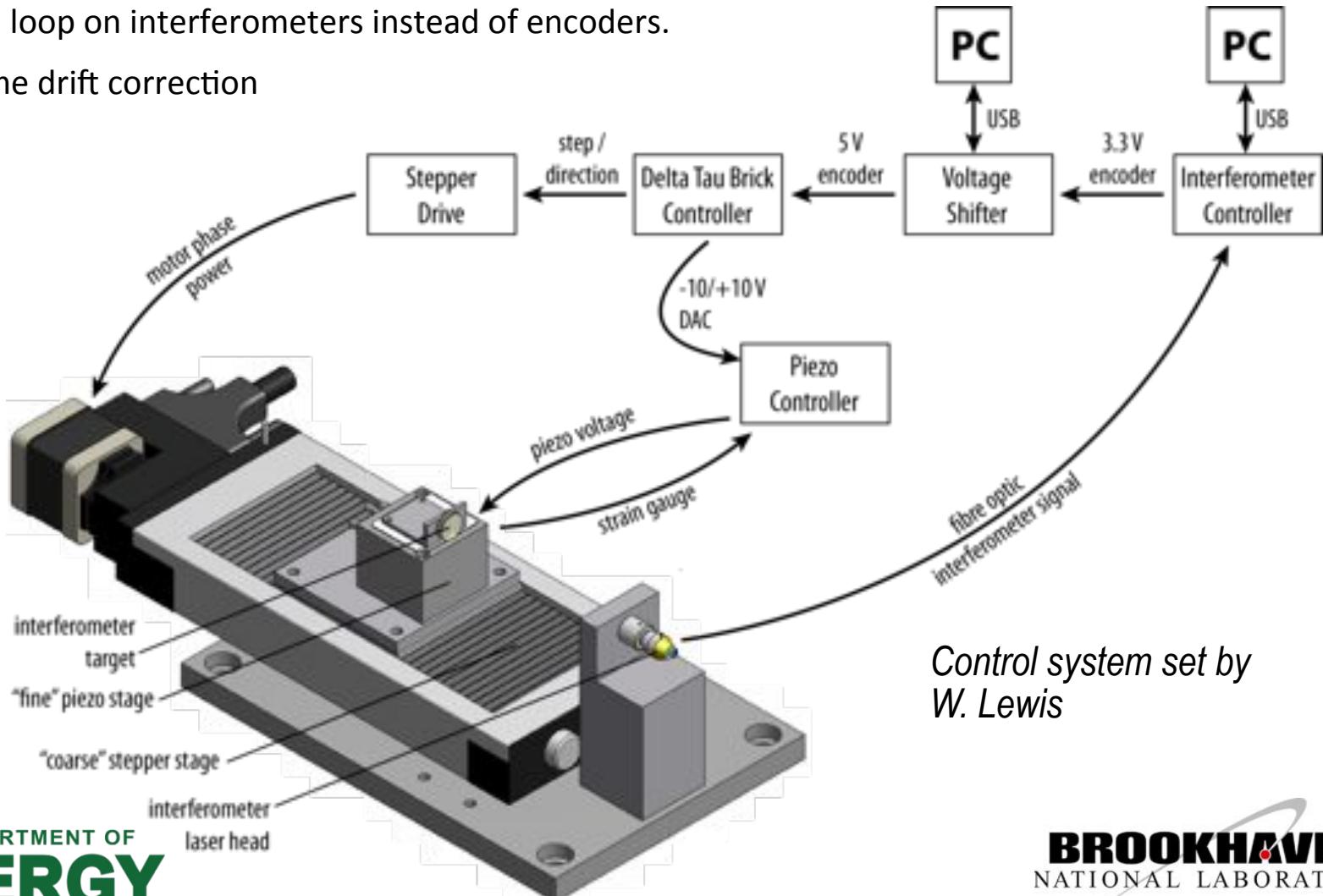
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Nano-positioning

✓ Closed loop between interferometers and fine stages

- Transfer the accuracy of the fine stages to the coarse stages during *on the fly* scans
→ closed loop on interferometers instead of encoders.
- Real time drift correction



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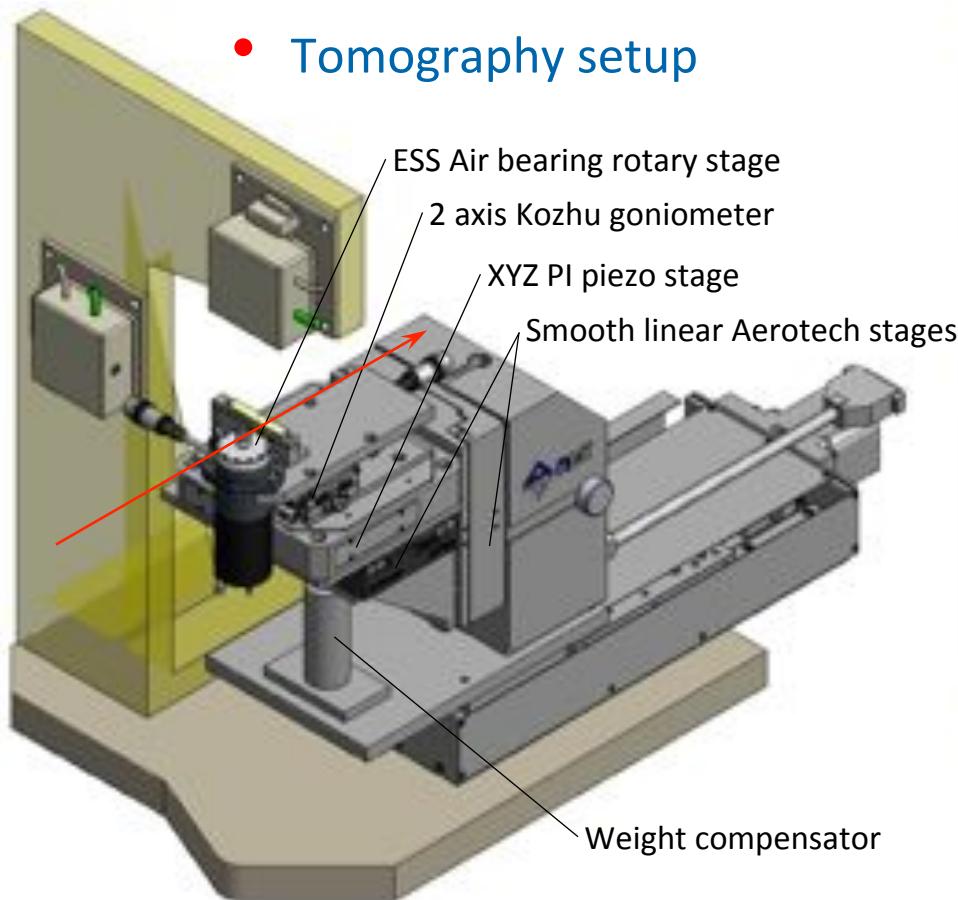
Nano-positioning

✓ Coarse stage: Aerotech ANT95-L (direct drive linear stage)

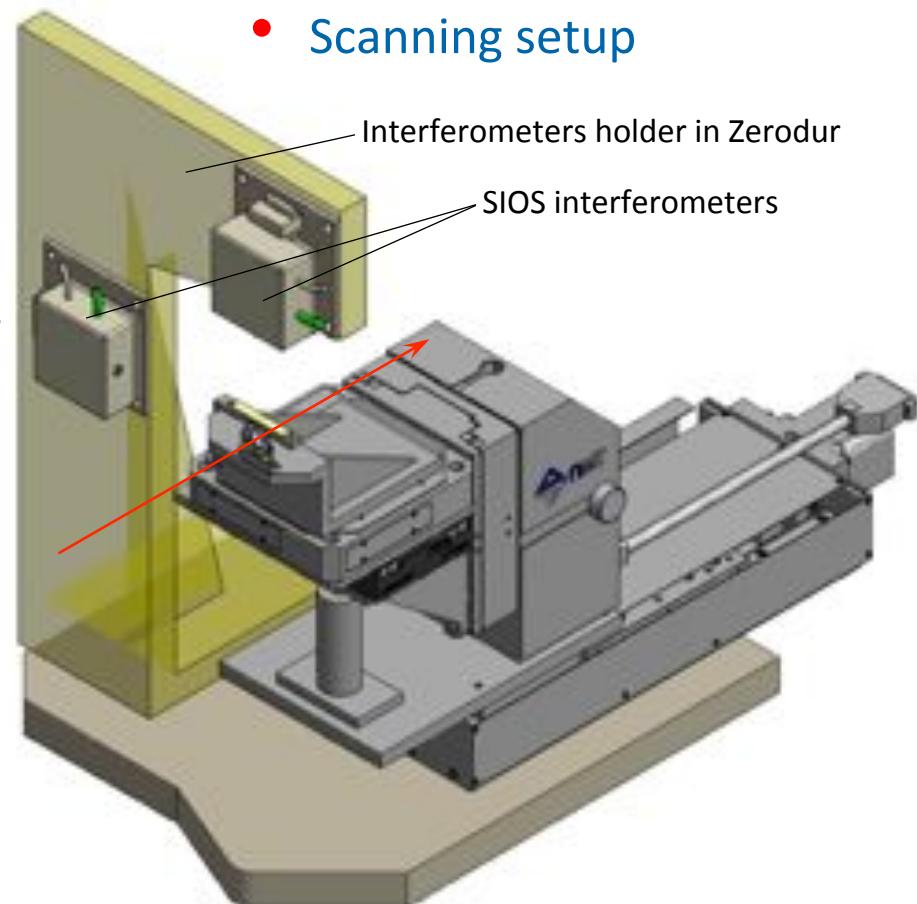
→ accuracy: ± 250 nm, bidir. repeatability: ± 75 nm



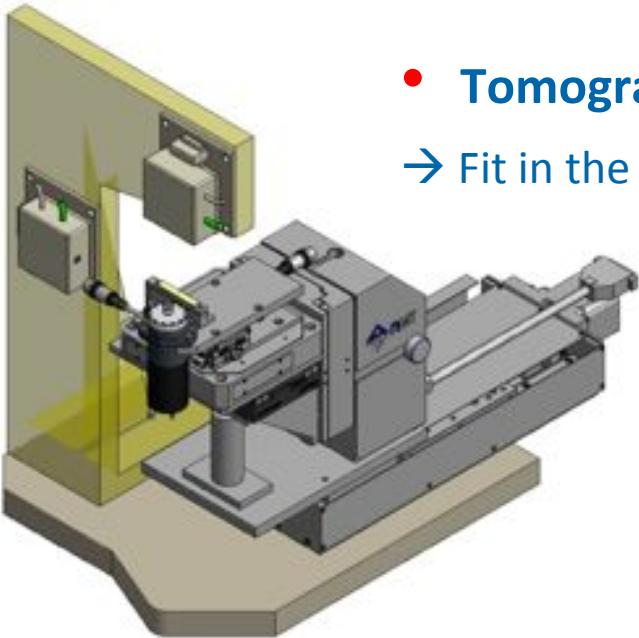
- Tomography setup



- Scanning setup

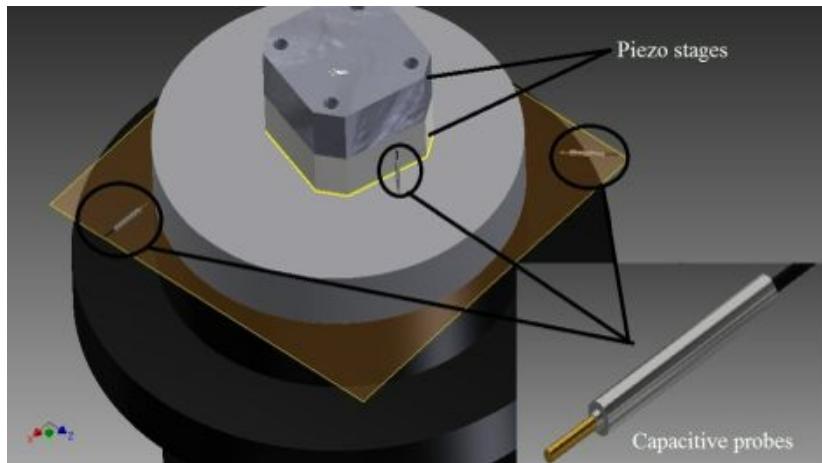


Nano-positioning

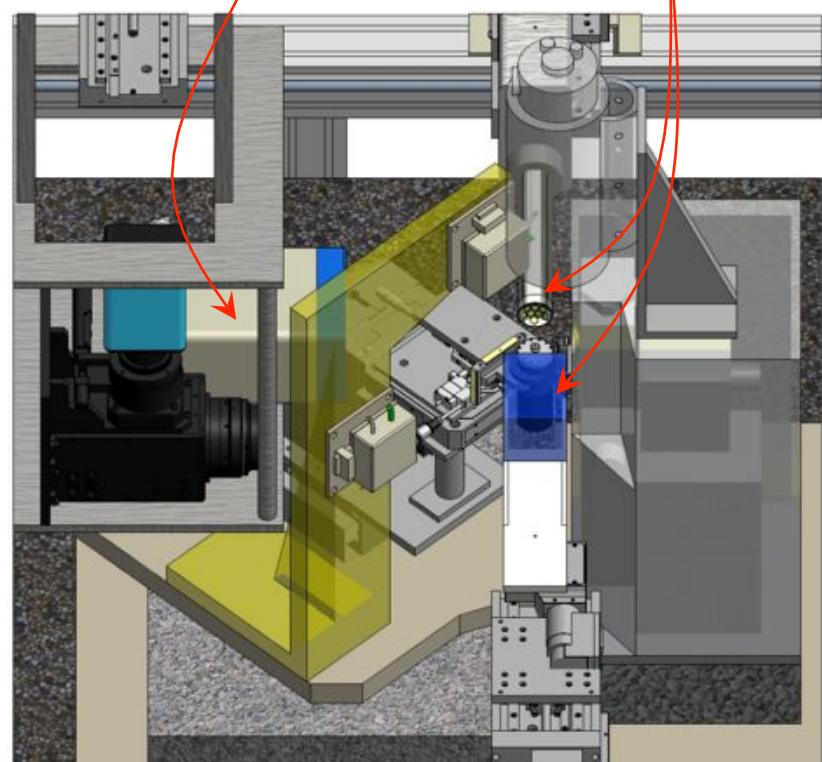


- **Tomography setup**
→ Fit in the 4 cm working distance of the nanoprobe

Project: dynamical correction of the rotary stage
run out for nanotomography acquisitions



- ✓ Simultaneous acquisition of fluorescence and diffraction tomography



Detectors strategy

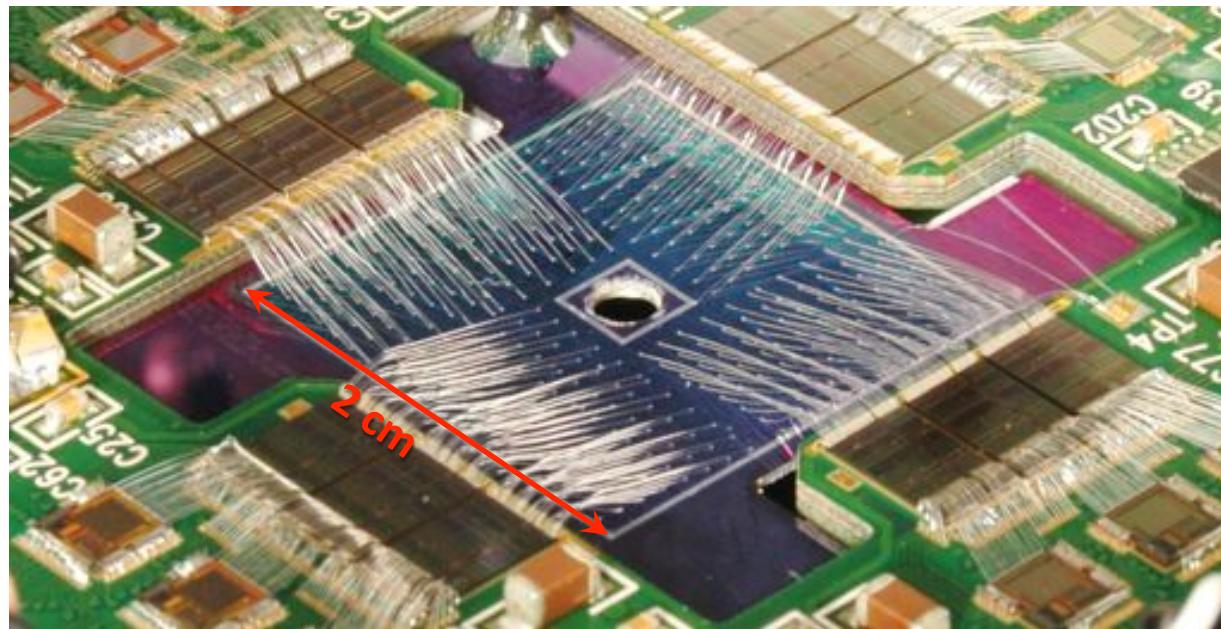
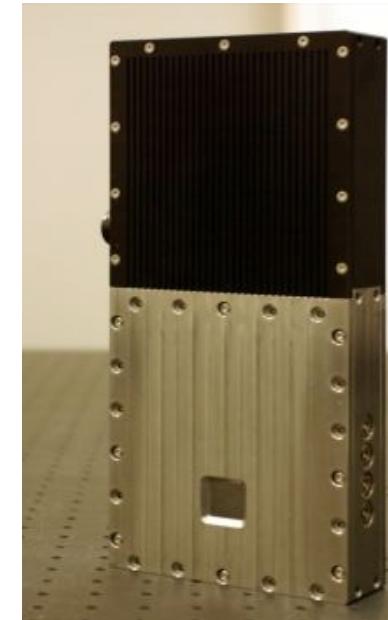
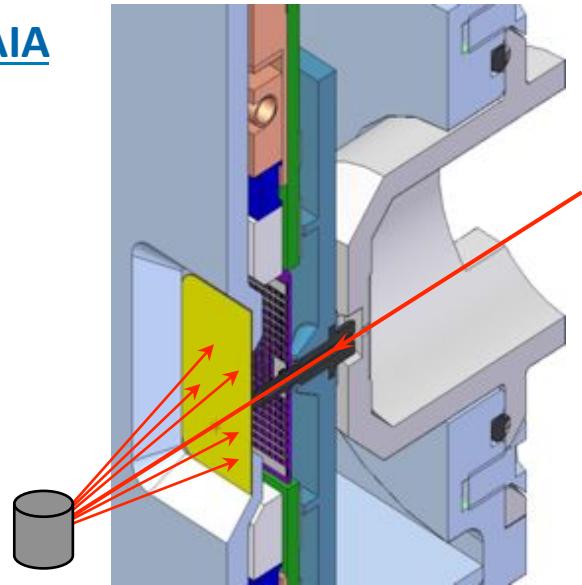
- Fluorescence Detectors
 - Energy Dispersive Spectrometers
 - SDD Vortex ME4 from SII & **MAIA (v.1 and v.2)**
 - Wavelength Dispersive Spectrometers
- Array Detectors
 - High resolution (full-field)
 - Diffraction

Detectors strategy: MAIA

✓ Energy Dispersive Spectrometers: Vortex ME4 from SII & MAIA

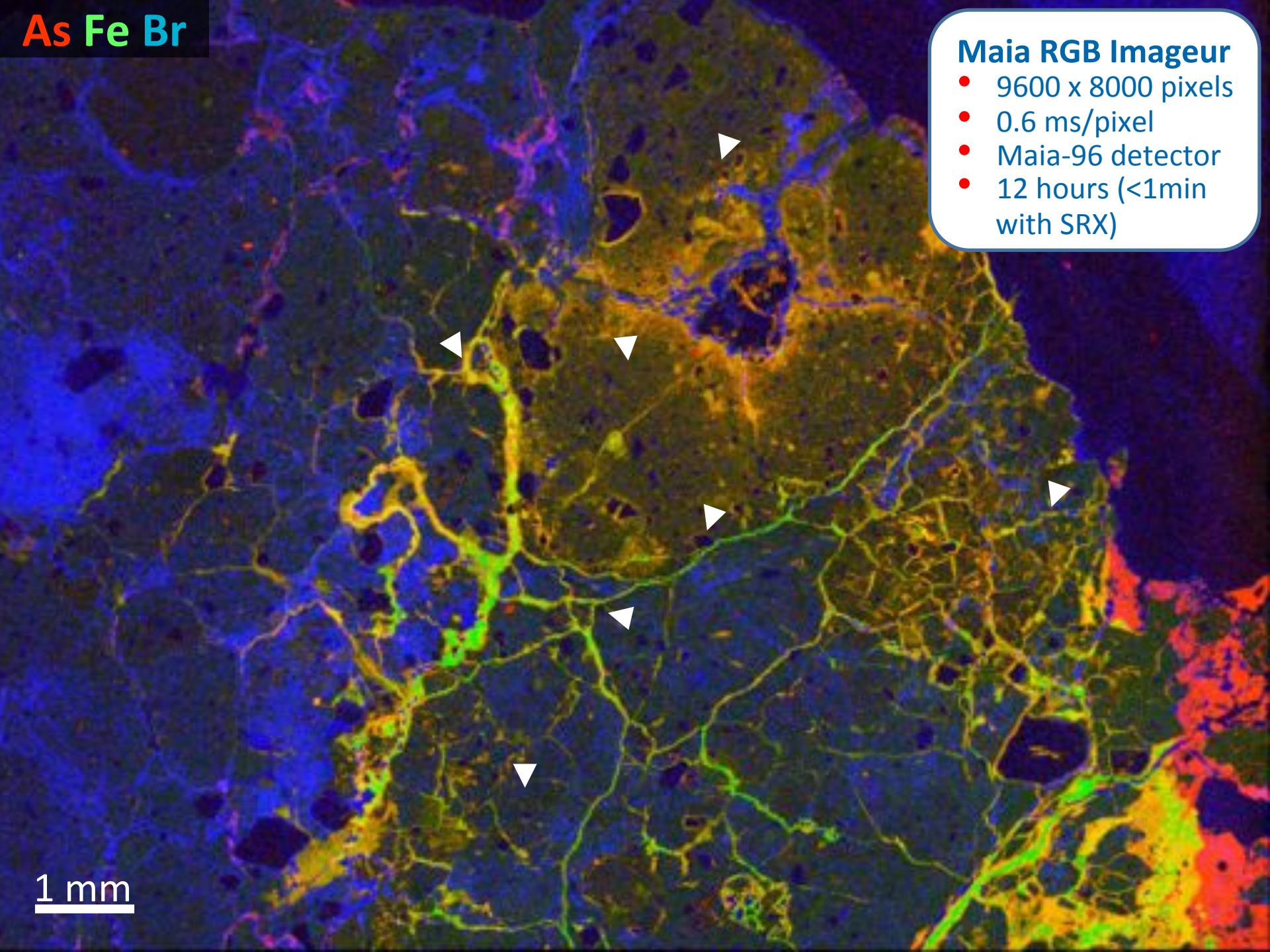
(BNL / SCIRO collaboration – P. Siddons, C. Ryan, R. Kirkham)

- MAIA version 1, back scattered geometry
 - ultrafast, drawback → bad energy resolution ~240 eV
- MAIA version 2, SDD detectors → x4 higher count rates,
~145 eV energy resolution



96 or 384 mm² photodiode detectors in a monolithic array, wire-bonded to 3 ASICS.

As Fe Br

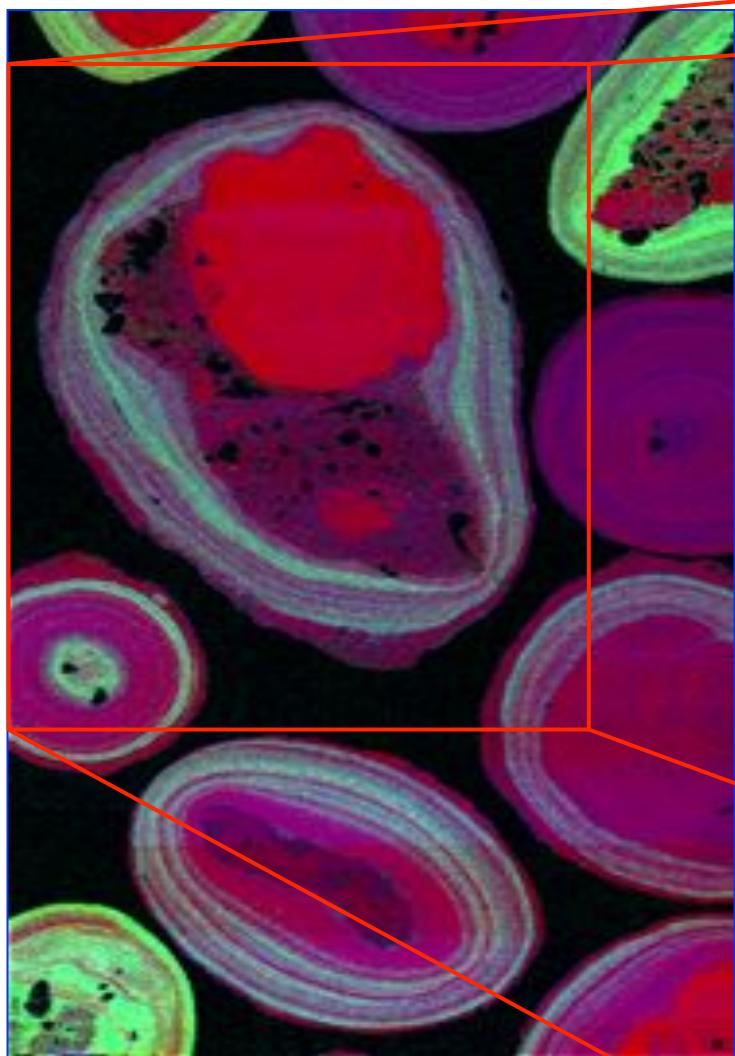


Maia RGB Imageur

- 9600 x 8000 pixels
- 0.6 ms/pixel
- Maia-96 detector
- 12 hours (<1min with SRX)

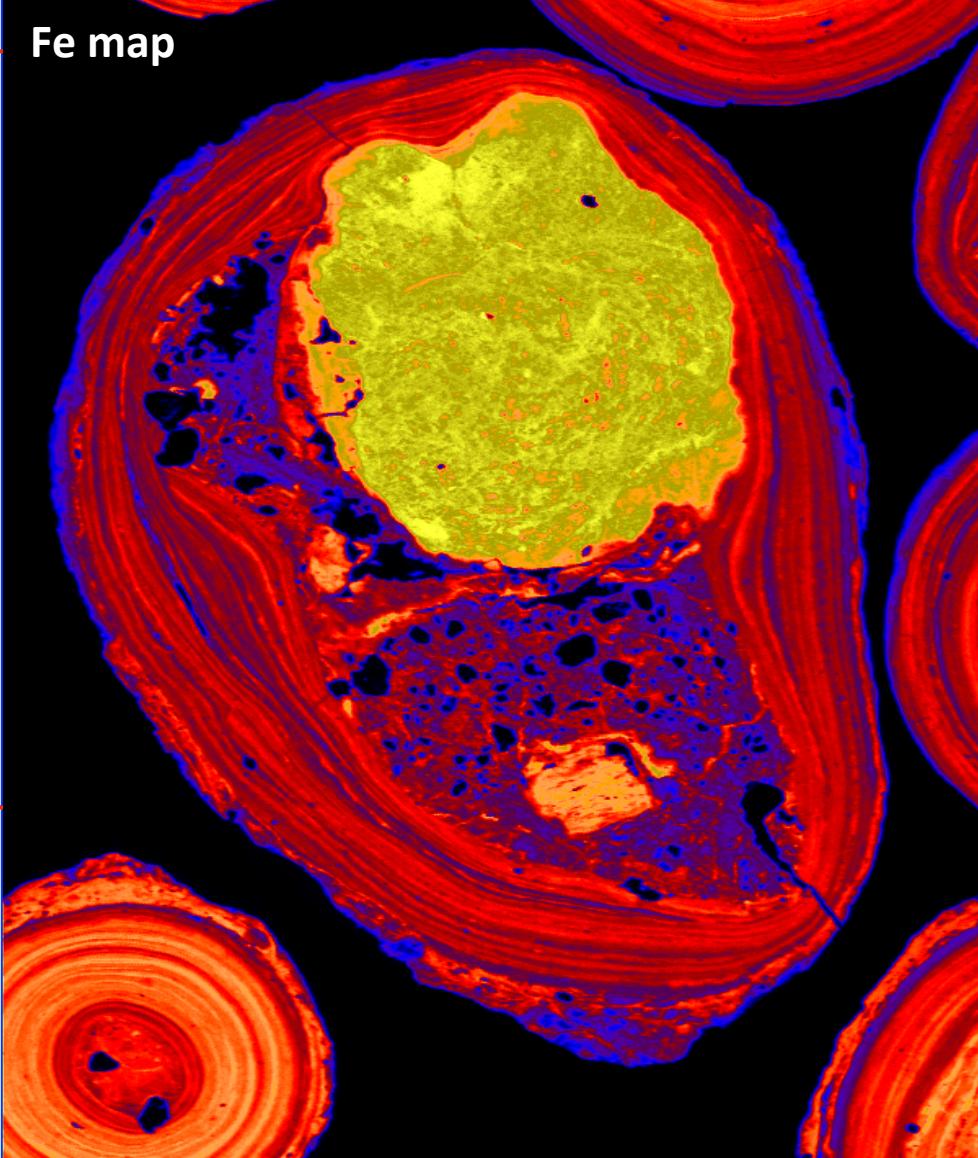
1 mm

Detectors strategy: MAIA



Fe-Y-Cu RGB composite

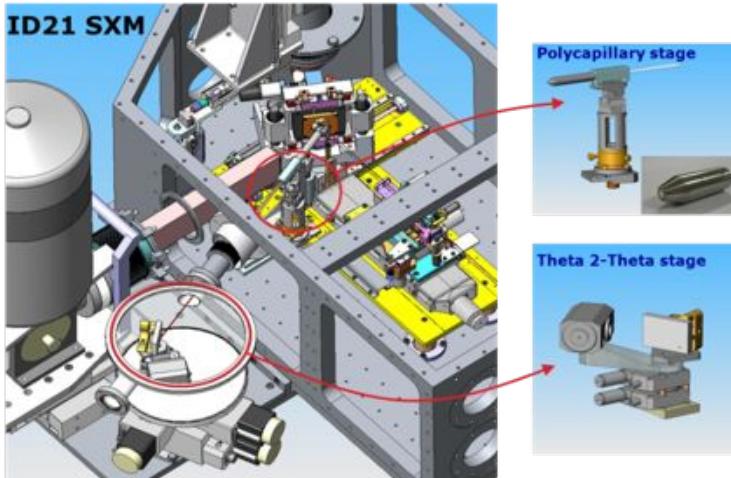
(1500 x 2624 pixel images, 13 x 21 mm²)



Detectors strategy: WDS

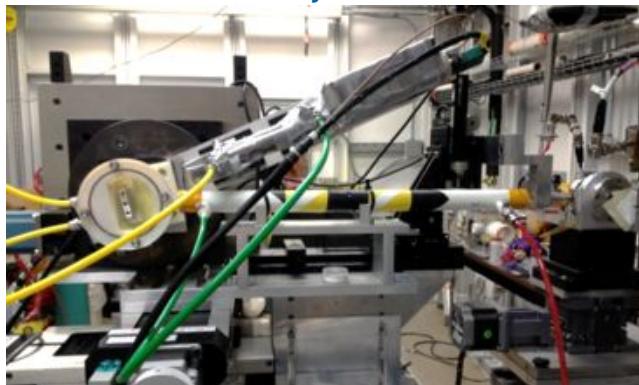
✓ LDRD project (*V. De Andrade, K. Attenkofer and J. Thieme*)

(a) Von Hamos and (b) High Efficiency Spectrometer – not yet funded...

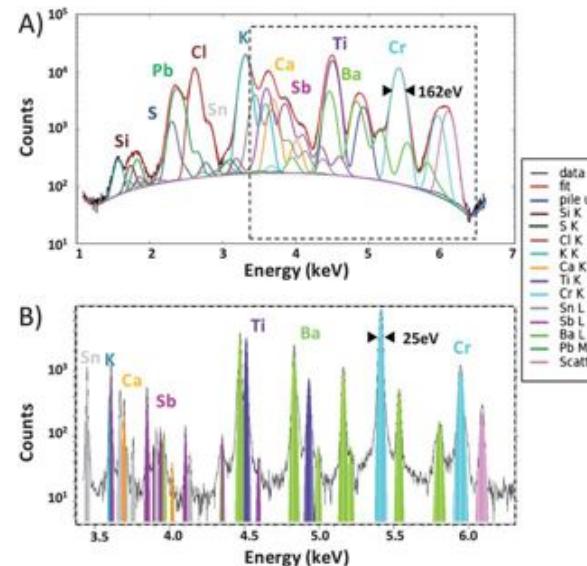


(figure courtesy J. Szlachetko et al.)

✓ LDRD project (*G. Seidler et al.*) – Funded
SRX team not directly involved



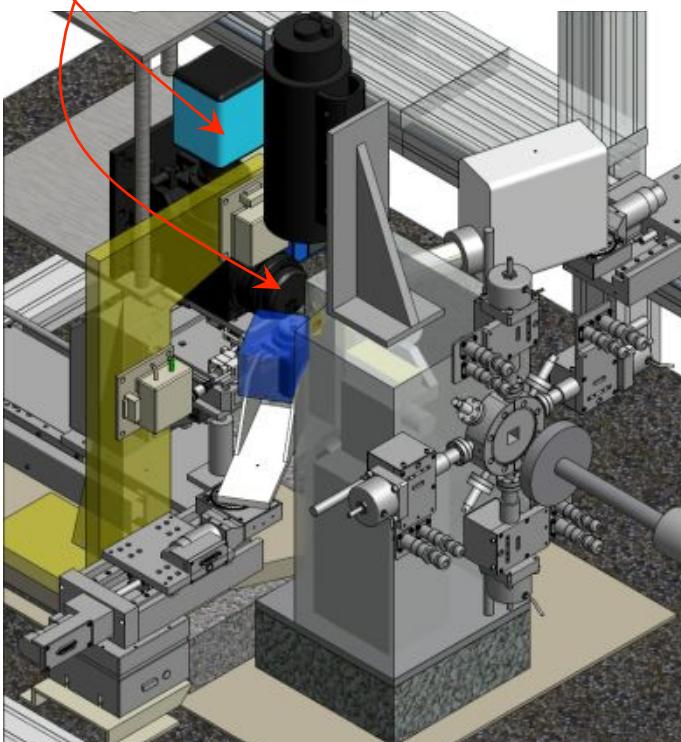
- High spectral resolution for emission lines separation
 - Combination of microscopy & X-ray Emission Spectroscopy
 - RIXS
 - X-ray Raman Scattering
- $10^{13} \text{ ph/s} \rightarrow \text{true asset for XES}$**



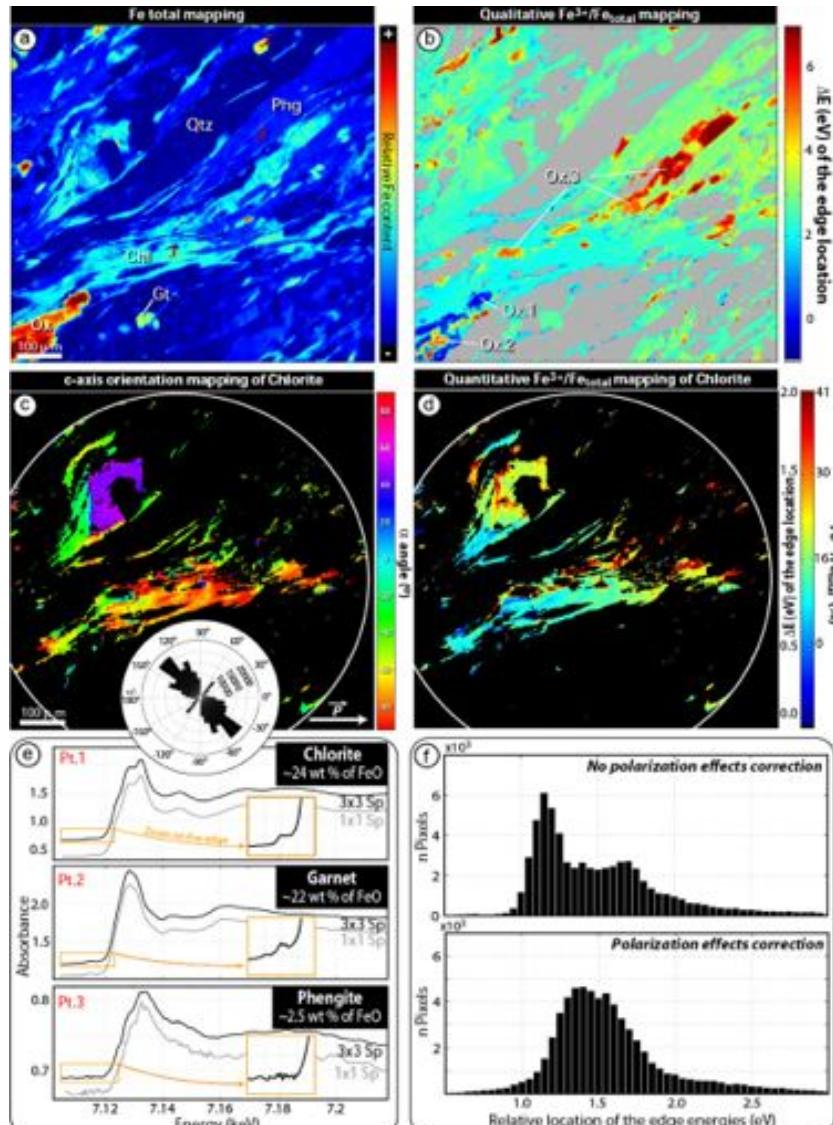
Detectors strategy: CCD array detectors

✓ High resolution detectors for full-field imaging - ultimate resolution ≈ 400 nm

- PCO edge (sCMOS Chipset) $\rightarrow 2k \times 2k$, 30 ms readout, 1.1 e⁻ noise, 27000:1 dynamic
- + 10 μm thick GGG:Yb scintillator (ESRF, T. Martin & P.A. Douissard)
- + 5X, 10X and 20X optics (Optique Peter assembly)



• Full-field spectroscopy

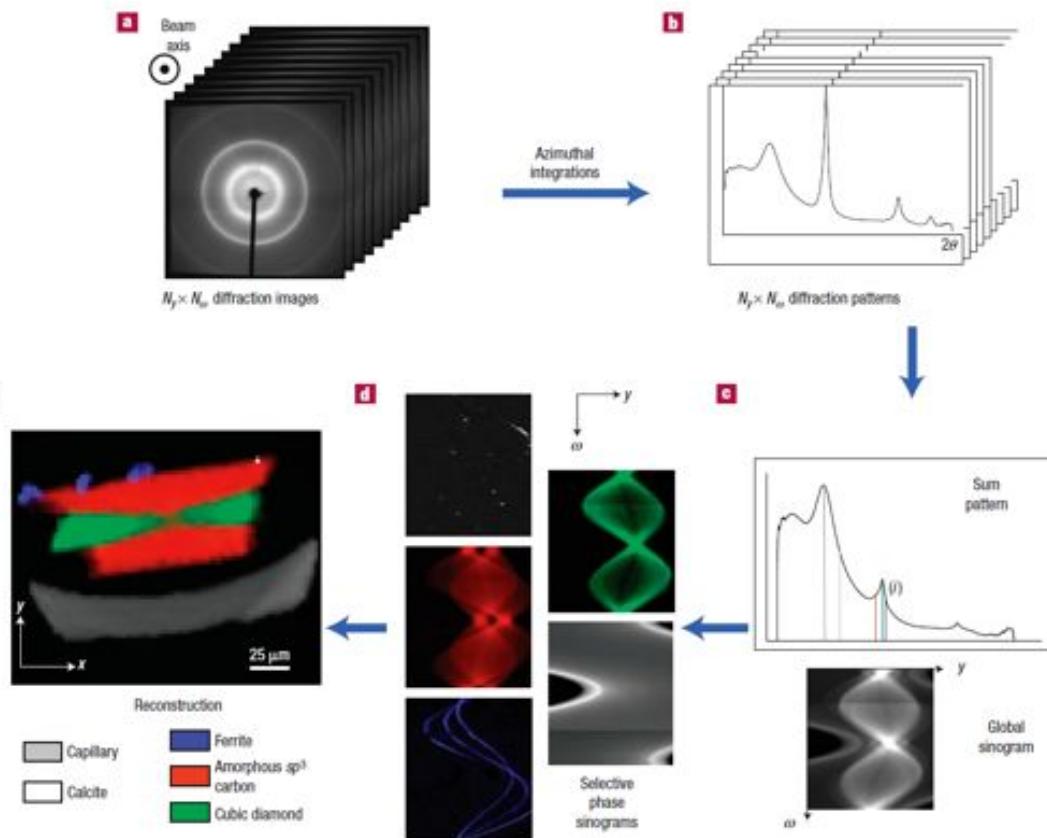


Detectors strategy: CCD array detectors

✓ Fast detector for Diffraction analyses

→ expected provider: Photonic Science

- 4k chipset (4 times 2k x 2k), very fast and low noise sCMOS chipsets
- Detection surface: $10.7 \times 10.7 \text{ cm}^2$
- Pixel size: $26 \mu\text{m}^2$
- Dynamic 27,000 : 1 (or 50,000 : 1 if binning)
- Readout: 30 ms



Phase identification of a C_{60} sample
crushed under pressure
(Bleuet et al., *Nature Material*, 2008)

ESRF, ID22, 18keV,
 $2.3 \times 2.3 \times 1.6 \mu\text{m}^3$ voxel
 10^{11} ph/s

Sample environment

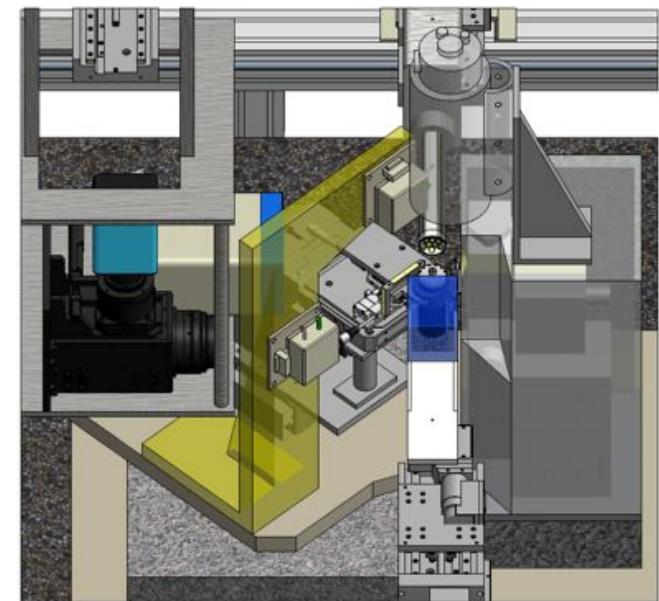
✓ **Microprobe:** 13 cm working distance

→ big volume press can fit in, as for instance the APS portable Paris-Edinburgh ($\varnothing 24$ cm)

✓ **Nanoprobe:** 4 cm working distance

→ ESS air bearing rotary stage can fit in

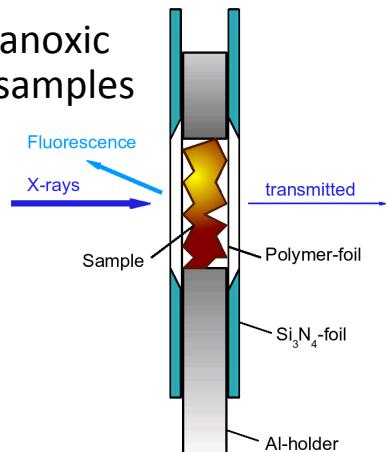
→ a Diamond Anvil Cell can fit in



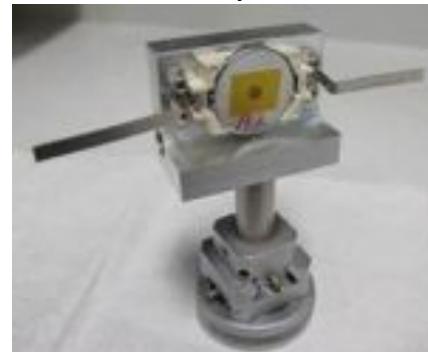
High resolution setup

What else? This workshop will help to specify the needs and fine tune the sample stage design

Cell for anoxic aqueous samples



Coin-type cell
for battery research



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NATIONAL LABORATORY
BROOKHAVEN SCIENCE ASSOCIATES

SRX Schedule / Conclusion

- **2010:** Preliminary Design Report
- **2011:** performing the specification docs for the main optical package and the KB systems
- **2012:**
 - contract awarding for the main optical package (Bruker) and the KB mirrors (WinlightX)
 - phase of design
 - purchase and test of End-station components
- **2013:**
 - equipment installation in the hutches
 - work on the sample stage
- **2014 (spring):** hot commissioning + *early science!* (@ 25 mA)